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TECHNIQUES
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PREFACE

The term "electronics" has come to mean many things to many people; in fact it would seem that nearly everything associated with electricity can be assembled under the term electronics. Possibly the term is taking on a new meaning. The American Standards Association defines it as follows: "Electronics is that branch of science and technology which relates to the conduction of electricity through gases or in vacuo." The term electronics in the title of this book and as used throughout its pages has that meaning.

The material covered has been directed to dealing with controlled beams of charged particles in vacuo or at very low pressures. The very large field of conduction of electricity in gases is not included.

During the last few years I have had the experience of training engineers in this work and in giving lectures to groups who were to use equipment requiring knowledge of the techniques. Although much of the contents of the book can be found at one place or another in the literature, I have attempted to include as many as possible of those techniques which are usually learned only by personal contact between laboratory workers or are acquired "the hard way" by much personal effort.

At first I felt that a complete reference to sources of material and equipment would be a desirable feature of this book. Since it is impossible to keep such references up to date, however, it has seemed best to direct the reader to the excellent buyers' guides now published annually. Only those references have been included which are required to supplement such a guide.

The historical background has been minimized as have unnecessary mathematical presentations, not because I think they are valueless but because their inclusion in the book does not appear to contribute to its utilitarian objectives. For the same reason, I

have tried to cull from the many references cited those which best carry out the intent of the book.

One of the major problems has been to set a dividing line between techniques for studying a subject and the study itself. For instance, experimental studies of electron optics are commonly carried out with the aid of electron optical devices. A similar problem is to differentiate between experimental and production techniques. I hope that the treatment of such problems will meet with approval.

In addition to the helpful discussions with many of my colleagues in the General Electric Company, I am grateful to Miss Dorothy Rogers and Miss Vicki Kalinowski for their cooperation in the preparation of the illustrations and manuscript and to Dr. Dushman for valuable discussions as well as some material from his forthcoming book, *Scientific Foundations of Vacuum Technique.*

C. H. BACHMAN

Syracuse, New York
September, 1947

CONTENTS

Chapter 1

VACUUM SYSTEMS GENERALIZED

1·1 Components Required

By the term vacuum system we shall denote any substantially closed system of tubes and chambers in which the pressure may be reduced or fixed at some value less than atmospheric. Unless otherwise specifically mentioned, this book generally will consider as a vacuum, pressures reduced to the value at which charged particles may be generated and moved around freely in space—their mean free path * is such that their behavior is not influenced to any extent by collisions with gas molecules. In the accepted sense of the term, the vacuum system should include some combination of the following:

> A working chamber, a pumping means, any gauges required, some means of controlling the pressure in the working chamber such as valves and variable leaks, provisions for adding gas; and in case it is necessary to bake out and thoroughly degas any of the components, an oven and high-frequency heating means.

1·2 Working Chamber and Accessories

The working chamber will be dependent upon the nature of the work and may consist of a bell jar, cathode ray tube or other type of tube, or glass or metal chamber in which are mounted the various elements with which to conduct the experiment. Usually these elements either will require the application of some mechanical motion which must be provided from outside the vacuum chamber or will have impressed upon them some electrical potential. This means that for each arrangement of parts, considerable ingenuity will be needed to get the desired

* The mean free path is discussed in sections 2·1 and 9·2.

mechanical motions. It is much easier to conduct the necessary potentials to the components since many types of insulated joints are available. If the chamber is to be sealed off after the work is completed, it is necessary to provide the proper constriction in the exhaust line so that the sealing-off process can be accomplished with a minimum detrimental effect to the vacuum.

1·3 Pumping System

It has become common practice in work of this kind to use two pumps, in series, to obtain the vacuum. The first is a mechanical pump which exhausts to the atmosphere and is used to pull the system down to a degree of vacuum such that its speed and efficiency decrease rapidly. Between this pump and the working chamber is placed a diffusion pump which ordinarily would not function at atmospheric pressure but begins to work in the pressure region in which the mechanical pump slows down. In general it can be said that mechanical pumps are used to remove the large quantity of gas at the start of the exhaust period and produce vacua down to a few microns. (See Table I for the relative values of pressure units.) Here the diffusion pump takes over and carries the vacuum on down to values which under ideal conditions may be as low as 10^{-9} or 10^{-10} mm Hg. For best results both as to pumping speed and ultimate vacuum, the ratings of both of the pumps should be well matched to the size of the working chamber and the degree of vacuum desirable. A wide choice in pump ratings is available. In case the type of work is not definite and may change from day to day, both as to volume to be exhausted and the quality of vacuum desired, it is desirable to provide a pumping system which will handle the greatest amount of the work most efficiently.

1·4 Vacuum Gauges

Although a number of terms are used in designating the degree of vacuum (see Table I), the two most common in this country are millimeters of mercury (mm Hg) and microns. It should be remembered that atmospheric pressure is roughly 760 mm Hg and one micron is $1/1000$ mm Hg. For low-pressure work in gases, it is quite customary to refer to the pressure as 1, 3, or 6, etc., mm Hg. As the pressure decreases to values below a few

TABLE I

Conversion of Pressure Units

	Dynes/sq cm (Barye, Microbar)	Grams/sq cm	Pounds/sq in	Atmospheres	Mm Hg (Tors)	Microns	Bars
Dyne/sq cm (Barye, Microbar)	1	$1\ 0197 \times 10^{-3}$	$1\ 4504 \times 10^{-5}$	$9\ 8692 \times 10^{-7}$	$7\ 5006 \times 10^{-4}$	$7\ 5006 \times 10^{-1}$	1×10^{-6}
Gram/sq cm	980 66	1	$1\ 4223 \times 10^{-2}$	$9\ 6784 \times 10^{-4}$	$7\ 3556 \times 10^{-1}$	$7\ 3556 \times 10^{2}$	$9\ 8066 \times 10^{-4}$
Pound/sq in	$6\ 8947 \times 10^{4}$	70 307	1	$6\ 8046 \times 10^{-2}$	51 715	$51\ 715 \times 10^{3}$	$6\ 8947 \times 10^{-2}$
Atmosphere	$1\ 0133 \times 10^{6}$	$1\ 0332 \times 10^{3}$	14 696	1	760	$7\ 6 \times 10^{5}$	1 0133
Mm Hg (Tor)	$1\ 3332 \times 10^{3}$	1 3595	$1\ 9337 \times 10^{-2}$	$1\ 3158 \times 10^{-3}$	1	1×10^{3}	$1\ 3332 \times 10^{-3}$
Micron	1 3332	$1\ 3595 \times 10^{-3}$	$1\ 9337 \times 10^{-5}$	$1\ 3158 \times 10^{-6}$	1×10^{-3}	1	$1\ 3332 \times 10^{-6}$
Bar	1×10^{6}	$1\ 0197 \times 10^{3}$	14 504	$9\ 8692 \times 10^{-1}$	$7\ 5006 \times 10^{2}$	$7\ 5006 \times 10^{5}$	1

This table gives the value of the pressure unit named at the left in the units named at the top. For example, 1 atmosphere is 760 mm Hg

tenths mm Hg, we usually change over to microns and speak of a few hundred or 10 or half of a micron. Then as the pressure decreases below about one-tenth of a micron we again refer to millimeters, as 10^{-5}, 10^{-7}, etc., mm Hg.

For the measurement of gas pressure in a vacuum system, a number of physical phenomena have been adapted. They include the pressure of a column of liquid, the pressure exerted by the motion of the gas molecules remaining in the system, the efficiency of the remaining gas to conduct heat away from an element in the vacuum, the amount of ionization obtained in the gas by a known amount of electron bombardment, and the damping effect on the vibration of a rigid thread in the vacuum system. Each of these phenomena has its own particular useful range of application and sensitivity and will be described in more detail later.

1·5 Pressure Control

It is quite often necessary to hold the pressure in a vacuum system at some value considerably higher than that to which the pump could take it if unrestrained. This may be done by using valves or constrictions between the pumps and the vacuum chamber or by the use of slow controlled leaks. It is also possible to achieve these results in some cases by actually developing the gas in the vacuum system by some such means as chemical or electrical.

1·6 Accelerated Degassing

To make possible a better vacuum, it is sometimes necessary to accelerate removal of the gas in and on the surfaces of the various elements which may be contained in the vacuum chamber and on the walls of the vacuum chamber itself. To accomplish this, various aids may be employed. The entire system may be baked at a high temperature by means of ovens which may be built about the system or may be lowered down over it by a system of pulleys and counterweights. It is also possible to accelerate degassing by merely playing a flame back and forth over the exposed surfaces until the temperature is raised sufficiently. Considerable benefit may be achieved at times, although not always, by holding the pressure for a time in the

region of a few mm Hg and applying potential in such a way that a violent gas discharge is obtained. This method often exerts a scouring effect, but since it may also result in increasing the amount of gas occluded on the walls, it cannot be used without reservation.

Suitably located and shaped conducting elements inside the system may be brought to extremely high temperatures by means of high-frequency induction heating. In certain instances, it is also possible to bring electrodes to high temperatures by the proper application of potentials which will cause excessive electron or ion bombardment. This effect is quite commonly seen in radio sets where, purposely or because of some fault in the circuits, one of the tubes becomes so loaded that the electron bombardment causes the plate to run bright red.

Chapter 2

PUMPS

2·1 General Discussion

A vacuum pump functions by creating a pressure gradient so directed that the gas of the vacuum system expanding to the region of reduced pressure is in turn caught by the pump and swept out the exhaust. At pressures just below atmospheric the laws of hydrodynamics may be applied to the gas flow, but at lower pressure values the phenomena tend to be molecular in nature and different relations exist. This molecular behavior becomes definite when the mean free path * of the gas approaches the order of magnitude of the physical dimensions of the system. Normally this occurs at a few microns and below. The ordinary mechanical positive displacement forepump operates most efficiently at the higher pressure while the vapor condensation pump depends upon molecular flow conditions for its operation.

Since the speed of a pump varies greatly with the operating pressure, it is desirable when choosing a pump to have knowledge either of the performance of the pump for different pressures or at least the speed at the pressure which will be encountered. This information is usually furnished by the manufacturers of the pump. A number of methods for measuring pump speed are available.[1] † Either the volume is held constant and the rate of change of pressure noted, or conversely the rate of change of volume at a constant pressure is determined. The most useful is the constant pressure method. An arrangement suitable for such measurement is shown in Fig. 2·1.

With the pump running, the needle valve or other suitable slow

* The average distance traveled by a molecule between collisions with its neighbors (see section 9·2).

† Superior numbers refer to the numbered references at the end of the chapter.

leak is adjusted to give the desired pressure as read by a suitable gauge at the pump inlet. A drop of mercury is then started through the calibrated capillary, and the volume it sweeps out in unit time is noted.

This volume (V_1) at atmospheric pressure (P_1) is converted to the equivalent volume (V_2) at the pump pressure (P_2), and the speed of the pump at that pressure is given by the relation

$$S = \frac{dV_2}{dt} = \frac{P_1 V_1}{P_2} \qquad (2\cdot1)$$

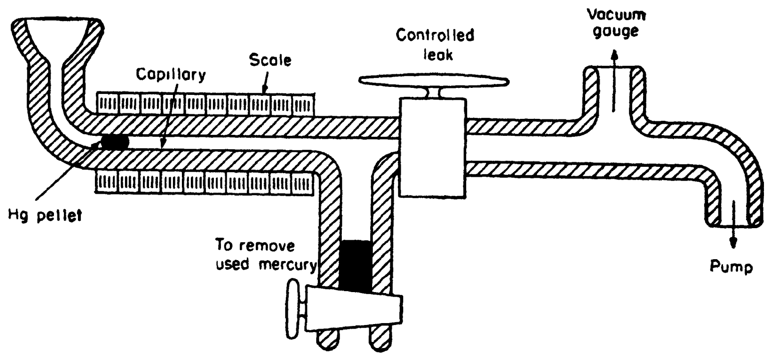


FIG. 2·1 Measurement of Pump Speed at Constant Pressure.

For an aperture connecting a region having perfect vacuum with one at some pressure Strong [2] finds that dry air at room temperature will diffuse into the vacuum region at the rate of 11.7 liters per sec per sq cm of aperture area. This value may be taken as a sort of upper limit for speeds of diffusion pumps, and on such a basis oil diffusion pumps attain factors of merit of about 0.5 as against less than half that for mercury pumps. A rough estimate of the speed of a well-designed pump may be made by determining the area of the jet clearance and applying a suitable factor of merit together with the 11.7 liters per sec per sq cm value of the "perfect pump."

An arrangement [3] with which more accurate speed values may be obtained and which is particularly useful in measuring high-speed pumps makes use of a mercury sealed chamber, accurately

calibrated and used in connection with a mercury manometer and a controlled leak.

More detailed consideration of gas flow phenomena and their effect on pump speed and conductivity of vacuum tubing will be found in the literature.[2, 4, 5, 6, 7] The problem of conductivity of vacuum tubing will be discussed further in Chapter 9. It should be stressed here, however, that the potentialities of a fast pump may easily be nullified by using it in conjunction with exhaust tubing of such a diameter or length that the flow of gas is restricted excessively.

2·2 Mechanical Pumps

(*a*) *Positive Displacement Types.* The mechanical pumps which produce the "rough" vacuum are usually referred to as "fore" pumps, "rough" pumps, or "back-up" pumps. Many makes are available, and they are furnished with pumping speeds varying from a few liters per minute to many hundreds of liters per minute and capable of producing in some cases lower than one-tenth of a micron. The larger capacity pumps are for the most part used in industrial applications.* Most common in the United States for use in connection with pumping systems on experimental set-ups are the pumps manufactured by the Welch Scientific Company and the Central Scientific Company. These pumps, although of quite different design, are somewhat similar in that they both consist of a rotary member and sliding vanes sealed in oil, and the pumping action depends upon actual physical displacement of the gas.

Figure 2·2 shows the Cenco Hyvac and Fig. 2·3 indicates the general structure of the Welch Duo-Seal type pump. The pumps furnished by these two companies give consistently good results if properly used. Performance curves are shown in Fig. 2·4. Both types of pumps are supposed to give vacua well under a micron under suitable conditions. Although such a pressure is many times quite satisfactory for experimental electronic work, it is very difficult to maintain these ideal conditions, and one can

* F. J. Stokes Machine Co., Philadelphia, Pa. Kinney Manufacturing Co., Boston, Mass.

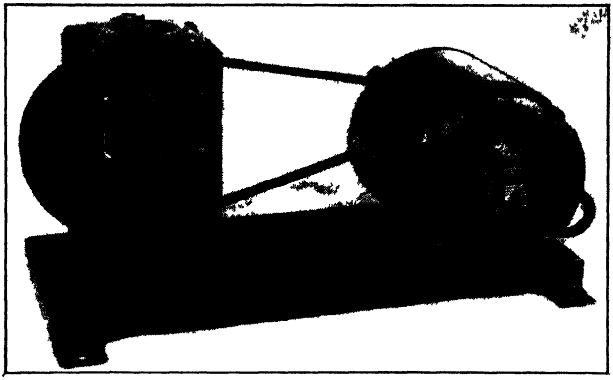


FIG. 2·2 Hyvac Pump. (Courtesy Central Scientific Company.)

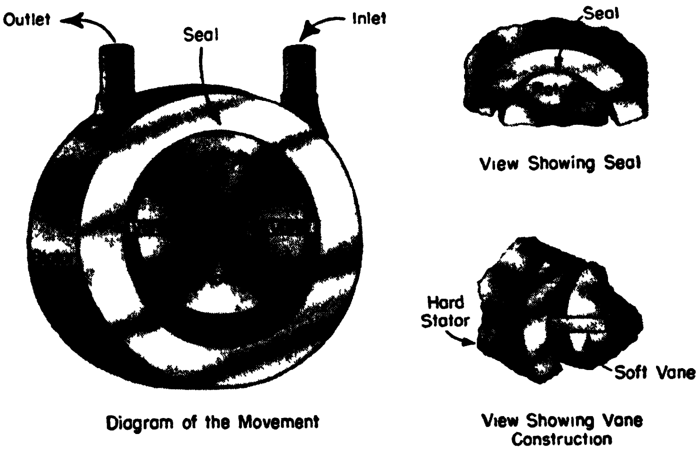


FIG. 2·3 Internal Arrangement of Pumping Mechanism of Welch Duo-Seal Type Pump. (Welch Scientific Company.)

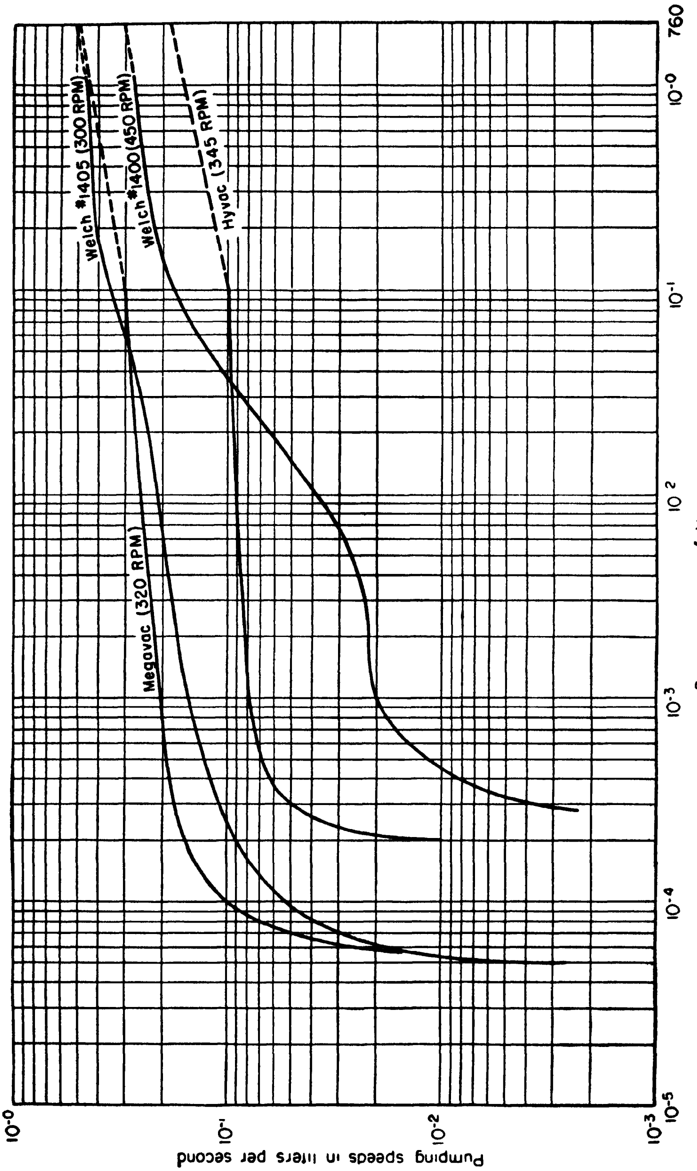


FIG. 2·4 Speed Curves of Mechanical Pumps.

consider himself fortunate if pressures of less than 10 microns are obtained consistently. This pressure is quite satisfactory when the pumps are used in connection with diffusion pumps, as most of these latter function quite well when exhausting into

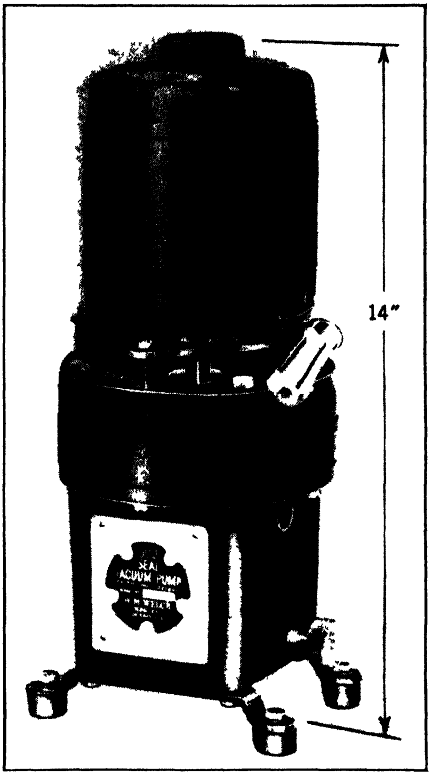


Fig. 2·5 Direct-Coupled High-Speed Mechanical Pump. (Welch Scientific Company.)

pressures up to the order of 50 microns. Both makes of pumps are usually driven with a V belt from a motor positioned on a metal base. A development of the Welch Scientific Company is the small directly coupled pump-motor combination shown in Fig. 2·5. The rated capacity (free air displacement) of this combination is approximately 10 liters per min and its operating

speed is 1750 rpm. It is of special value in applications where the vibration, weight, and space requirements of the other type pumps cannot be tolerated.

It should also be mentioned here that the compressor of the ordinary domestic refrigerator can sometimes be used as a fore-pump. In some cases, it is possible to obtain pressures as low as 30 to 50 microns with such a pump. One difficulty encountered with them is that they tend to vaporlock, and in such instances, the pressure suddenly rises to several hundred microns which, of course, may stop the diffusion pump. Although it is possible to rebuild them with suitable by-passes and stronger valve springs, it is usually not economical in view of the other pumps available. They are mentioned here because there are some pumping applications where a few hundred microns is acceptable. Some of the large-capacity diffusion pumps operate satisfactorily under these conditions.

(*b*) *Molecular Type.* One type of mechanical pump developed rather early, as well as some subsequent variations, makes use of one member rotating at very high speeds (many thousand rpm) within a very close-fitting case. The drag of the rotating member on the gas molecules causes them to follow grooved paths which are placed so as to cause a continuous flow through the pump. The Gaede pump and the Holwecke pump make use of grooved revolving cylinders while another model uses a disk with spiral grooves cut in its sides. All these pumps must be exhausted to a back pressure of a few microns and are capable of reaching vacua of the order of 10^{-6} mm Hg.

2·3 Molecular Vapor Pumps

The development in molecular diffusion pumps has taken place rapidly in the past 30 years and has been an important contributing factor in connection with much of our modern physics research. Many materials have been tried as the vapor material. For some 15 years, mercury was by far the best working medium, but recent years have seen the development of a number of oils which it seems will soon nearly replace mercury. These oils may be obtained with vapor pressures at room temperatures as low as 10^{-8} mm Hg. (See Table I.)

TABLE I

CHARACTERISTICS OF DIFFUSION PUMP FLUIDS

Supplier	Oil Type	Vacuum Attainable in mm Hg
J. G. Biddle Co., Philadelphia, Pa.	Apiezon oil A	10^{-5}
	Apiezon oil B	10^{-6}
Eitel McCullough, Inc., San Bruno, Calif.	Eimac pump oil	approx. 3×10^{-7}
Distillation Products, Inc., Rochester, N. Y.	Butyl phthalate	4×10^{-5}
	Butyl sebacate	2×10^{-5}
	Amoil	7×10^{-6}
	Amoil-S	2×10^{-6}
	Octoil	2×10^{-7}
	Octoil-S	5×10^{-8}
	Myvane DP-20	5×10^{-6}
Dow Corning Corp., Midland, Mich.	Silicone fluids No. 702 and No. 703	5×10^{-8}
National Research Corp., Vacuum Engineering Division, Boston, Mass.	Type TS diffusion pump oil	2×10^{-5}

Compiled from the suppliers' literature.

With them it is possible to operate without a refrigerant, whereas in the case of mercury which has a vapor pressure of about 10^{-3} mm Hg at room temperature, it is necessary to use a refrigerant such as CO_2 or liquid air to prevent mercury vapor from contaminating the system.

Although many types of structures of diffusion pumps are available on the market, the fundamentals of their operation are shown in the typical designs of Fig. 2·6. The vapor from a heated boiler passes through a jet arranged so that gas from the vacuum chamber tends to diffuse into the pump vapors before they condense on the walls of the pump. These gases mingling with the high-velocity vapors are given an impetus in the direction away from the vacuum chamber and are removed by

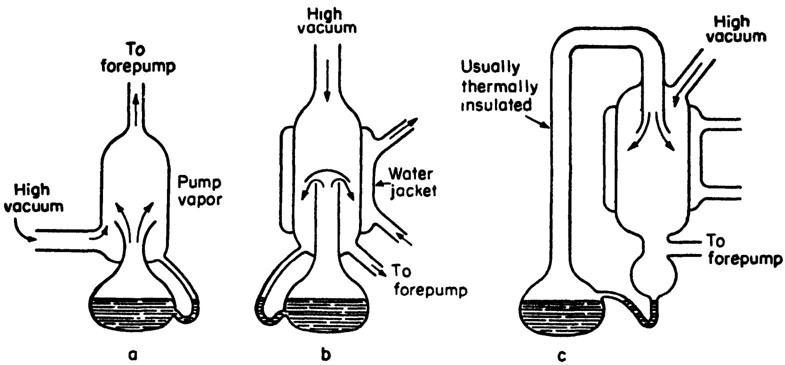


FIG. 2·6 Molecular Vapor Diffusion Pump Types.

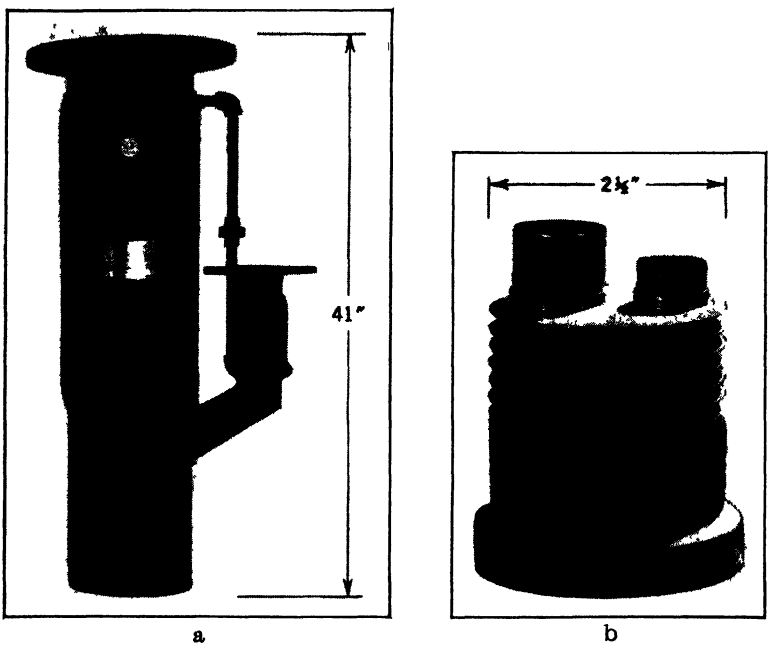


FIG. 2·7 Typical Metal Oil Diffusion Pumps Available Commercially. (*a*) Type H-6 Diffusion Pump Having Capacity of 3000 Cubic Feet per Minute (12,700 Liters per Second). (Courtesy of National Research Corporation.) (*b*) Type VMF-2 Oil Diffusion Pump Having Capacity of 2 Liters per Second. (Courtesy of Distillation Products, Inc.)

means of the forepump while the condensed pump vapors circulate back to the boiler to complete the cycle.

The design variations commercially available have mostly to do with physical sizes of the pumps, the number of jets in series, and the physical arrangement of these jets.* Some of the pumps are metal throughout, others are all glass, and some are combinations in which the main chamber is in glass, but the carefully designed jets are of metal or plastic. Both mercury and oil

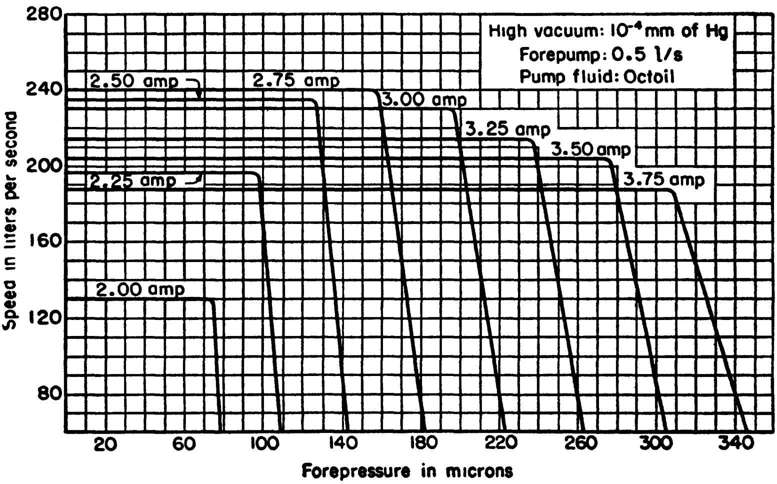


FIG. 2·8 Speed-Forepump Characteristic for Vapor Pump Type GM220AB, Distillation Products, Inc.

pumps are available with liquid or air cooling. One commercial type of pump features a construction designed to keep the pump oil purified by fractionation and the volatiles and tarry residues segregated by thermal diffusion to a special chamber. The range of sizes of commercial pumps may be seen in Fig. 2·7, where (*a*) is a large (41 in. high) metal water-cooled pump of 3000 cu ft per min (1270 liters per sec) capacity made by the National Research Corp. (type H-6), and (*b*) is a small air-cooled pump

* Distillation Products, Inc., Rochester, N. Y. National Research Corp., Vacuum Engineering Division, Boston, Mass. Eitel McCullough, Inc., San Bruno, Calif. General Electric Co., Cleveland. Ohio. Litton Engineering Laboratories, Redwood City, Calif.

with a speed of about 2 liters per sec supplied by the Distillation Products, Inc. (type VMF-2).

Although gas flames were originally most commonly used in connection with mercury pumps in laboratory installations, oil pumps call for rather good temperature control, and the use of electric heaters, either internal or external, is rapidly becoming the rule for all pumps. Typical performance curves for a vertical

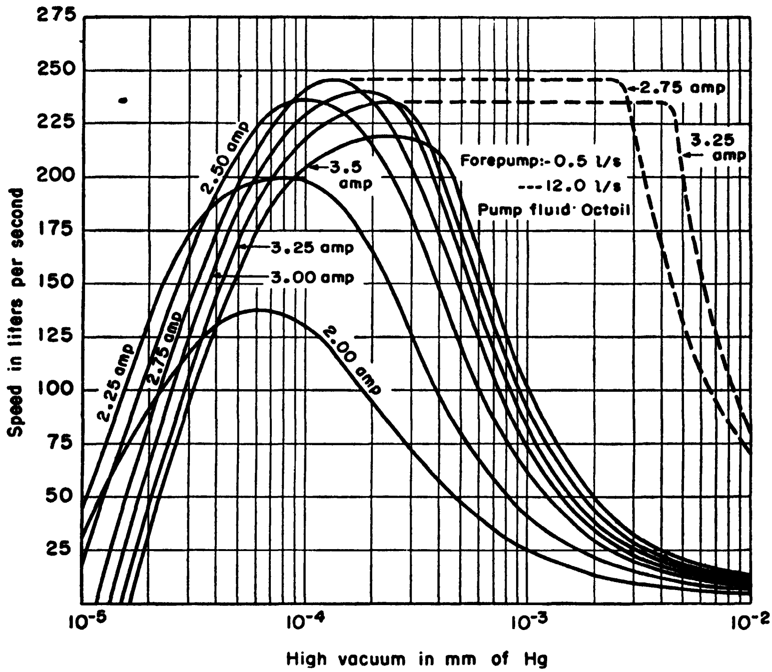


FIG. 2·9 Speed-High Vacuum Characteristic for Vapor Pump Type GM220AB, Distillation Products, Inc.

multiple-jet "umbrella" type diffusion pump are shown in Figs. 2·8 and 2·9. The effect of forepump speed upon the diffusion pump performance is shown by the dotted lines of Fig. 2·9.

2·4 Aspirators

Although not of practical use as a main pump for electronic work since it does not provide sufficient vacuum, another type of

pump which is quite common is the aspirator. This device which may be used alone or which sometimes aids in backing up a diffusion pump makes use of the Venturi effect. Figure 2·10 shows that high-velocity liquid or vapor is caused to pass through a constriction which actually assumes the shape of a jet. The reduced pressure at the point of this constriction creates a vacuum in the adjacent volume which is limited to a value approximately the vapor pressure of the liquid used. A common example of the aspirator is that which is attached to the faucet of the regular water supply. Vacua of several mm Hg may be obtained by this means.

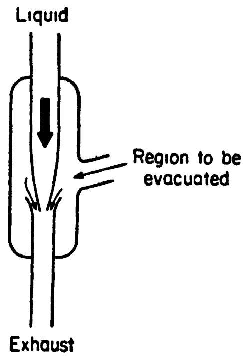


FIG. 2·10 Water Aspirator.

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Chapter 3

TRAPS AND BAFFLES

3·1 Mechanical and Electrical Baffles

No vacuum pump available is capable of producing a complete vacuum. Even under the most ideal operating conditions, there is always a considerable amount of gas remaining in the vacuum chamber. This residual gas is free to travel back and forth through the chamber, down the vacuum line, through the pumping system, and out to the atmosphere. It is true, of course, that the pumps act as a considerable barrier for such journeys, but it is a fact that the diffusion of gas even at the most extremely high vacuum is such that, for instance, some of the vapors from the pump will in time be found in all parts of the vacuum system. One of the problems of a good vacuum system is to keep this diffusion to a minimum, and one of the ways of doing it is to create a mechanical or an electrical obstacle in the path of the diffusing gases and still not restrict too much the main flow of the gas which is being pumped out.

When such a vacuum is attained that the mean free path of the gas is of the order of the diameter of the tubing connecting the pump and the vacuum system, the result may be that a molecule of gas will collide with the wall a number of times on its way out. As the vacuum is reduced still lower, the molecule may be able to travel in a straight line over a considerable portion of the evacuating line before meeting any obstacle.

It has been found that under these latter conditions, proper placement of a mechanical obstruction such as a bend in the pipe or a series of alternately arranged baffle plates will reduce considerably the diffusion of "residual" gas from the relatively high pressure region near the pump where the vapors are hot to the remoter parts of the vacuum system. Figure 3·1 shows

typical baffle arrangements. In such cases, the vapor upon striking the obstacle will condense instead of moving on into the vacuum chamber. Such baffles are commonly cooled, usually with circulating water.

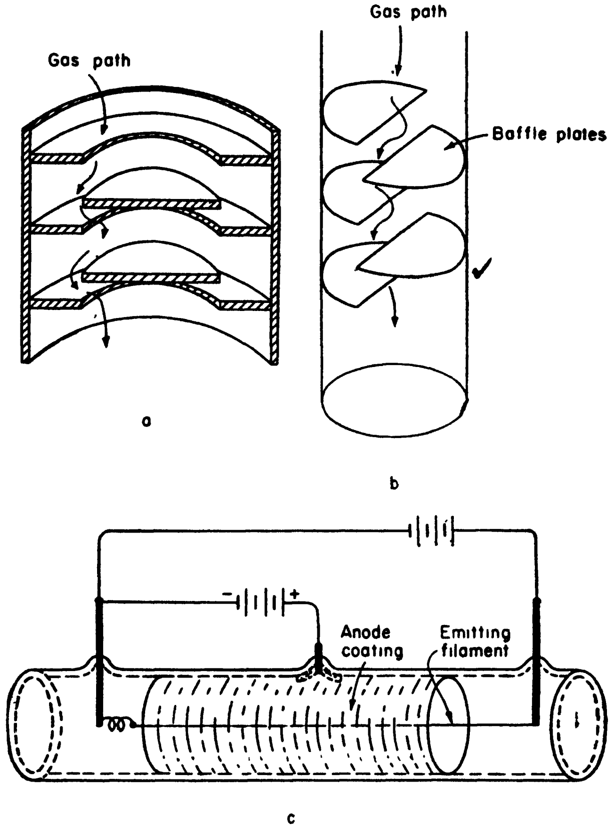


FIG. 3·1 Baffle Arrangements.

Another method of stopping this diffusion is to place an array of electrically energized electrodes in the vacuum line in such a way that the gases coming into the region are ionized and drawn to collecting plates having the proper potential. This system is not used extensively.

3·2 Refrigerants

Another arrangement to impede the progress of gas back into the vacuum system, and as a matter of fact to aid in removing gas from the vacuum system itself, is to place in the system a region of extremely low temperature. The problem here again is to present a surface to the interior of the vacuum system such that a maximum of condensing surface is available for a minimum of constriction to the regular pumping process. In addition it must be arranged so that the surface is maintained at some greatly reduced temperature by the use of an externally ap-

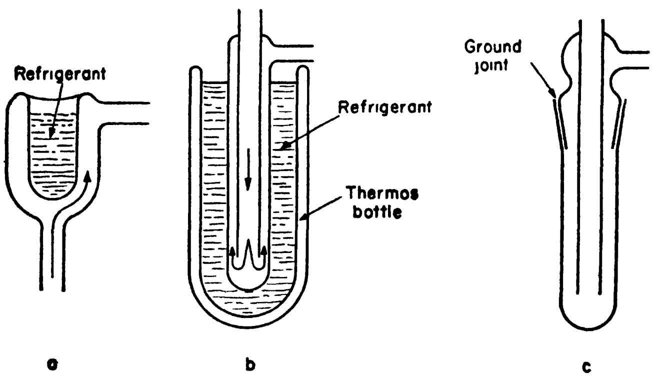


Fig. 3·2 Types of Refrigerated Traps.

plied refrigerant. The trap should be constructed so that inherently it has efficient mechanical baffling action. It is desirable that no gas molecule go through the trap without touching a cooled area at least once. Additional factors involved in the construction of such traps take into account the ease with which the trap is supplied with refrigerant and is cleansed of condensed material after the prolonged periods of operation. A number of good trap designs are shown in Fig. 3·2.

For optimum conductivity of the traps *b* and *c* Dushman and Lafferty have found that the inner and outer tube diameters should be in the ratio of 0.62 when the glass thickness is negligible. The commonly accepted practice of keeping equal cross-section areas for the inner and outer paths gives conductances of about 90 per cent of the optimum.

Cleaning can be accomplished by physically removing the trap or by arranging to bake it or flame it out while on the pumps. If this cleaning is not taken care of, a trap can actually become a source of contamination to the system, a so-called virtual leak. Depending upon the gas or vapor which is to be removed from the system, a number of refrigerants may be used. They are shown in Table I.

TABLE I

TEMPERATURES OBTAINABLE WITH VARIOUS REFRIGERANTS

Refrigerants	*Temperature Attained, °C*
NaCl 1 part and snow or fine ice 3 parts	−21.3
Solid CO_2	−78.51
Alcohol and CO_2 (solid)	−72.0
Chloroform and CO_2 (solid)	−77.0
Ether and CO_2 (solid)	−77.0
Liquid air, boiling point	−192
Liquid nitrogen, boiling point	−195.8
Liquid oxygen, boiling point	−183

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Liquid air rarely maintains its identity as such during ordinary storage as the liquid nitrogen having a lower boiling point tends to evaporate faster, leaving an excess of oxygen. For this reason liquid air becomes more dangerous after prolonged storage. Particular precaution should be taken in connection with its application where it might come into contact with oils or greases. This is especially true of its use in traps for vacuum systems using oil diffusion pumps.

When liquid air is poured into a glass container such as a trap or thermos bottle, the precaution should be taken to reduce the container temperature slowly by pouring a few drops at a time and agitating the container until danger of thermal shock is reduced.

Where it is not advantageous to obtain solid CO_2 commercially in cake form, simple expansion equipment is available for use with tanks of liquid CO_2. With these machines small cakes may be made up quite rapidly when desired. Solid CO_2 must be broken up for use in traps. It is very convenient to have available for this purpose a small sack or piece of cloth of very durable quality such as duck. The CO_2 cake is wrapped in this cloth and may then be pounded or crushed with a minimum loss of the material.

Table II gives the vapor pressures of water and mercury at various low temperatures.

TABLE II

VAPOR PRESSURES OF WATER AND MERCURY

Material		*Approx. Pressure, mm Hg*
Water	10C	9.2
Water	0C	4.6
Water	−78C	6×10^{-4}
Water	−98C	2×10^{-5}
Mercury	20C	1×10^{-3}
Mercury	0C	2×10^{-4}
Mercury	−30C	5×10^{-6}
Mercury	−78C	3×10^{-9}

Handbook of Chemistry and Physics, pp. 1721, 1722, 1728, Chemical Rubber Publishing Co., Cleveland, Ohio, 1941–1942.

3·3 Purifying Lines

Arrangements whereby the gas in the system may be brought in a controlled fashion into physical contact with desired materials may be used for several purposes. One is to remove the gas for the purpose of obtaining a better vacuum. In other cases, preferential removal of one or more of the gaseous components may be desired for the purpose of obtaining a purer remaining gas. A number of phenomena may be applied.

The use of activated charcoal is quite common, its action being such that gas to the extent of many times its volume may be absorbed, and also its porous properties are such that a high degree of efficiency of mechanical baffling is achieved by pumping

through it. This is accomplished, however, at considerable cost to the pumping speed. When the charcoal becomes contaminated it is possible to reactivate it by the application of heat. In some installations, by the use of electrical heaters embedded in the charcoal, this reactivation is accomplished without removing the charcoal from the system.

Another material used to remove water vapor and oxygen is finely divided copper. Here the action is accelerated and efficiency is increased by maintaining the copper at high temperature. Many materials are available for the removal of water vapor (see Table III). These materials depend upon absorption, adsorption, or both for their action.

TABLE III

EFFICIENCIES OF DRYING AGENTS

Drying Agent	Residual Water Vapor after Drying—mg per liter
Trap at liquid air temperature	1.6×10^{-23}
Phosphorus pentoxide, P_2O_5	$<2 \times 10^{-5}$
Magnesium chlorate, $Mg(ClO_4)_2$	$<5 \times 10^{-4}$
Conc. sulphuric acid, H_2SO_4	3×10^{-1}
Silica gel	3×10^{-2}
Calcium chloride (anhydrous), $CaCl_2$	1.4 to 3×10^{-1}

International Critical Tables, Vol. III, pp. 385, McGraw-Hill Book Co., New York, 1928.

J. Yarwood, *High Vacuum Techniques*, 2nd Edition, p. 72, John Wiley & Sons, New York, 1945.

Another scheme which is very useful in certain specific applications where there are traces of mercury in the system is to present surfaces of sodium or potassium to the gas. For instance, metallic sodium may be distilled into the system and made to condense on a considerable wall area. The amalgam formed by the sodium combining with the mercury very effectively reduces the amount of mercury present.

In all these arrangements of traps and baffles, regardless of whether it is some chemical action, condensation, or adsorption which does the work, it is essential that the surface presented to the gas at low pressure be kept as great as possible while the path followed by the gas being exhausted under higher pressures is kept sufficiently large to provide good pumping.

Chapter 4

VACUUM GAUGES

4·1 General Discussion

There is much misunderstanding and lack of comprehension in connection with the use of gauges on vacuum systems. The idea is altogether too prevalent that a gauge connected anywhere on the vacuum system will give an accurate picture of the condition in the system. Actually the situation is something like trying to obtain the temperature of the interior of a house with a thermometer on a cold day. If the thermometer is placed close to a window or next to the bottom of a door, the temperature reading will be low. It will be in general much lower near the floor than near the ceiling. The thermometer would not be placed in the oven or close to the furnace outlet to get a value which represents the conditions in the house. As a matter of fact, the conditions in the house cannot be accurately given by one reading of the thermometer in any case. Similarly, a gauge placed in the vacuum line near the pump will not give a true picture of the vacuum system any more than a gauge placed in some remote section of the vacuum system, possibly near a pinhole leak or some contamination which is constantly evolving gas.

Some types of gauges have considerable time lag in their operation so that by the time the gauge has responded and has been read, conditions in the vacuum system have changed. In the McLeod gauge, for instance, a small sample of the gas is taken from the system, and that sample of gas is representative of the main volume at that instant only. If the pressure is fluctuating or changing for any reason, conditions may have changed markedly by the time the McLeod has been adjusted and read. Furthermore, great care must be taken in using any one of the gauges to insure that it does not serve as a source of error itself. For instance, an ionization gauge which operates at elevated

temperatures may be so badly contaminated that it will give readings that are too high because of gas evolution within itself.

It should be emphasized, therefore, that a gauge on a vacuum system is not something which can be taken for granted like a steam gauge or a time clock. If accurate values are to be obtained with such an instrument, great care must be used. On the other hand, where relative values are sufficient, which is often the case, it is not quite so essential to take great precautions. The gauges to be described here constitute the more commonly known ones; some can be classed as absolute gauges and some as secondary. The absolute gauges are those which need not be calibrated against a standard. It is possible to calculate their response to given degrees of vacuum. All the secondary gauges require calibration. Many interesting variations of the principles used by these instruments have been developed.[1, 2, 3] Table I shows the ordinarily accepted useful ranges of a number of gauges.

TABLE I

APPROXIMATE RANGE OF USEFULNESS OF VACUUM GAUGES

Type	Upper Limit (mm Hg)	Lower Limit (mm Hg)
McLeod	1	10^{-5}
Knudsen	10^{-3}	10^{-6}
Quartz fiber	10^{-2}	10^{-5}
Ionization gauge	10^{-3}	10^{-7}
Thermocouple	10^{-1}	10^{-4}
Pirani	10^{-1}	10^{-4}

4·2 Absolute Gauges

(*a*) *McLeod Gauge.* The McLeod gauge, in addition to being one of the oldest gauges,[4] is probably one of the most common and useful. Basically it consists of an arrangement of glassware and mercury such as is shown in Fig. 4·1 with which it is possible to trap a known volume (V_1) of gas from the vacuum chamber at pressure (P_1). This gas sample is segre-

gated from the system and compressed by raising the column of mercury. This mercury column customarily is raised so that the level in the matching side capillary comes opposite the top of the gauge capillary. Thus a rather large volume of gas at low pressure is compressed into capillary tubing of small bore. The amount of this tubing required to contain the gas under the standard compression conditions is measured by the difference

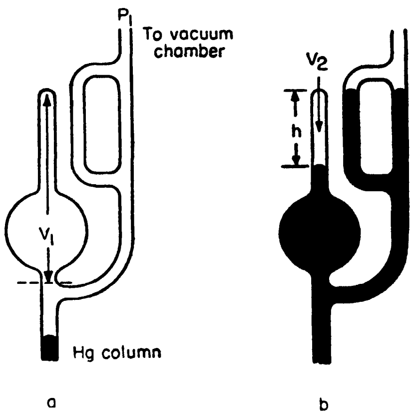


FIG. 4·1 Operating Principle of McLeod Gauge.

(h) of the two mercury levels, and since the bore is constant and of known value (V_2) is available. The original pressure can be calculated from the relation $P_1V_1 = P_2V_2$, where (P_2) is indicated by the height of the mercury column (h). In actual practice the calculating is done in advance and a scale made up so that observations of the height of the mercury in the capillary tube can be read directly as pressure (P_1).

The mechanism of trapping off the known volume of gas is accomplished by raising a column of mercury past the tube leading from the vacuum system. This may be accomplished by any one of a number of methods (Fig. 4·2). One (a) is manually to raise a mercury reservoir connected with the bottom of the gauge by a flexible hose. Another (b) makes use of a plunger and a cylinder which, when pushed down, drives the mercury up the column. In another (c), atmospheric pressure drives the

mercury to the desired height while another source of vacuum (very rough) is required to lower it when a new reading is to be taken.

A number of precautions are necessary in the operation of this instrument. First, the capillary, and in fact all parts of the gauge including the mercury, must be kept very clean. Also mercury must be free of any gas or water vapor. For instance,

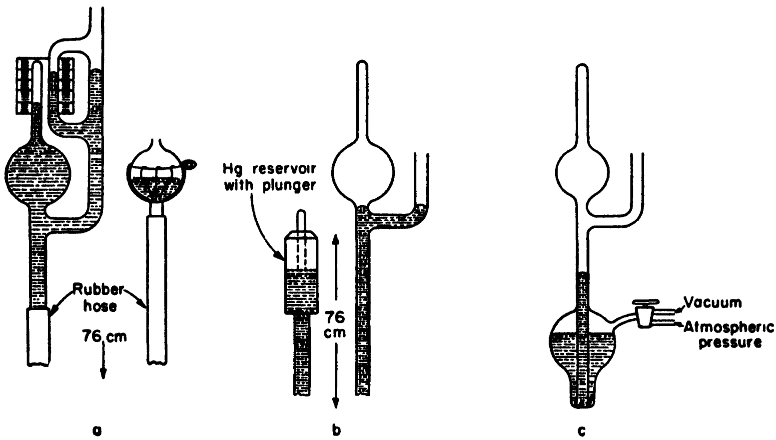


FIG. 4·2 Means of Controlling Hg Height in McLeod Gauge.

if fresh mercury is placed in a gauge, it is quite possible that small amounts of gas will be trapped in it and carried up into the trapped-off volume when the mercury column is raised, thus giving inaccurate values. Warming the mercury and considerable raising and lowering of the mercury column will usually clean this condition quite satisfactorily.

Care should be taken that when the mercury is up in the gauge air should not be allowed to enter the main vacuum system. If this happens, lowering of the mercury in the gauge will usually result in a violent action which may break the glass and drive mercury throughout the system. A McLeod gauge should always be operated in a rather slow and deliberate manner, particularly when the mercury is entering a constriction of next smaller size. Sometimes the mercury in the capillary tubing will become broken into segments, which usually may be brought down by tapping

the capillary. If this fails, a soft flame carefully played along the glass will usually take care of the situation.

Gentle tapping of the capillary, as with a pencil, is also advisable when taking a reading where the mercury nearly fills the capillary. Often the mercury will not follow the glass walls perfectly so that the tapping will aid it in attaining its proper place. Under conditions of very good vacuum the mercury will apparently completely fill the capillary, and when the compressing column is lowered it will drop away from the end of the capillary only after considerable lag. It is said to be "sticking," and the condition is commonly referred to as a "sticking vacuum." The value at which this occurs depends upon the capillary bore diameter and its cleanliness. In general it requires a vacuum of 10^{-5} mm Hg or better. The McLeod gauge does not indicate compressible gases or vapors.

(*b*) *Knudsen Gauges.* In this type of gauge, use is made of the momentum possessed by the gas molecules in their random motions. Molecules heated by a properly placed element are caused to impinge upon a delicate vane hung by a suspension similar to that used in galvanometers. The magnitude of the vane deflection obtained by this method depends upon the number of gas molecules in the space, and this makes possible the measurement of the degree of vacuum. The gauge, which is independent of the type of gas, was first described by Knudsen [5] and has been made more practical by Dumond and Pickels.[6] This gauge is fairly complex to construct and is probably useful only under laboratory conditions. When properly used it is capable of very good results and is sensitive in the region below a micron.

(*c*) *Quartz Fiber Gauge.* A vacuum indicator proposed by I. Langmuir,[7] and used only infrequently in ordinary electronic laboratory work, makes use of the attenuation of vibration of a quartz fiber as a result of the viscosity of the gas, the coefficient of viscosity varying with the pressure below a few hundred microns. An arrangement is made to set the fiber in vibration and observe its damping characteristic. It is possible to calculate the sensitivity of this device, or it may be calibrated with some other instrument. Its sensitivity is confined to the region be-

tween 10^{-2} to 10^{-4} mm Hg and differs in its response to gases of different molecular weights.

(*d*) *Manometers.* Simple manometers are of little value in measurement of extremely low pressures. They are, however, very useful where pressures at or above a few millimeters of mercury are encountered, such as when introducing gas under controlled conditions to a vacuum system. These manometers may be either of the open or the closed type, and although mercury has been quite commonly used, oil gives a sensitivity roughly fifteen times greater. Care must be taken in the use of oil to insure correct readings.

A disturbing property of oil is its readiness to absorb large quantities of gas. Thus, if an open-tube manometer is arranged on a vacuum system with the open end exposed to the air, it may be found that the gauge reading will vary with the passage of time. This is due to the absorption of air at the open end of the gauge and its resulting passage through the gauge oil and into the vacuum. When this characteristic is recognized and allowed for, it is quite possible to obtain good results with these oil gauges.

4·3 Secondary Gauges

(*a*) *Ionization Gauges.* The ionization gauge [1, 8, 9] is probably the one most used in connection with work in the pressure region below about a micron. Above this value its action becomes very erratic because of multiple ionization, but when used in its proper range it has a straight-line characteristic. The gauge is a triode usually mounted in an envelope similar to a radio tube and contains a cathode, a grid structure, and a collecting plate. The grid structure, which is actually the anode, is usually operated positive to the cathode by about 150 volts. Surrounding the grid structure is the collecting plate, which is run negative to the cathode by some such voltage as 22½. Figure 4·3 shows a workable circuit in about its simplest form.

In principle, the gauge measures gas pressure by collecting and measuring the positive ions formed in a space by an electron beam of fixed current and velocity. The electrons leaving the cathode, in numbers controlled by varying the filament tem-

perature, are accelerated toward the grid by the positive potential applied there. The desired emission is adjusted as indicated by measurement of the current received at the grid. Because of the open structure of the grid, a proportionate number of electrons will not be collected and will pass into the region between the grid and the collecting plate. The number of positive ions formed which are due to collisions of these electrons with the gas molecules in this space is a function of the gas pressure of the system. These ions are collected by the plate and measured usually with a microammeter.

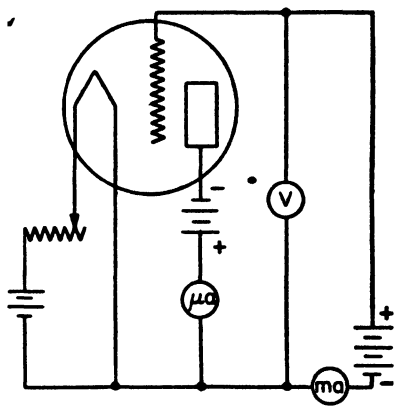


FIG. 4·3 Simple Circuit for Ionization Gauge.

Multiple ionization is not encountered, provided the pressure is low enough, so that below about a micron, the characteristic is a straight line. Thus, for calibration of the gauge in this straight-line region it is essential only that two points of known pressure be used.

A number of types of triodes have been used for these gauges. In addition to the many commercial models available, certain of the standard radio tubes of the earlier vintage are fairly satisfactory. For extreme sensitivity and accuracy, however, a number of precautions should be taken in the design of the ionization gauge. Long leakage paths should be arranged in the structure so that very small leakage currents will not affect the readings, which may be as low as 0.01 μa. Because of the high operating temperatures associated with it, causing gas evolution from its parts, this gauge may give erroneous readings.

It is essential for obtaining a true reading that the entire gauge be well outgassed and allowed to come to a stable operating condition. In gauges where the three elements are metal electrodes mounted as in a radio tube, the electrodes may be outgassed by

one of two methods. The collector plate may be bombarded with electrons so that it will come to a good red temperature, or a high-frequency induction coil "bombarder" may be arranged external to the tube and the induced currents used to raise the temperature.

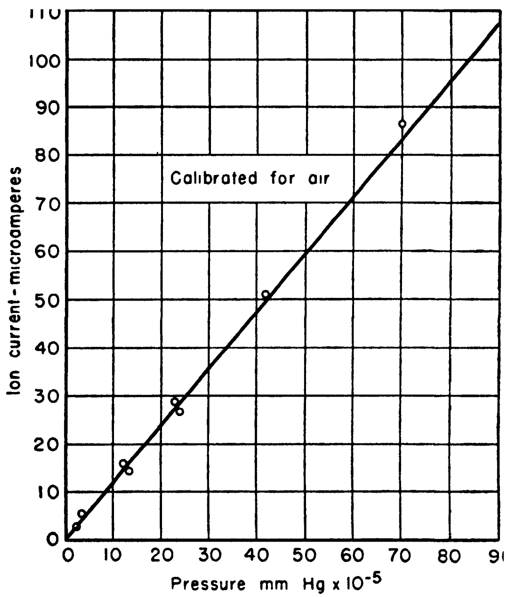


FIG. 4·4 Typical Ionization Gauge Characteristic Curve. (Distillation Products, Inc., Gauge VG-1A.)

Another type of gauge [10] makes use of a reversed spiral grid with both leads brought outside the envelope so that the grid wire may be heated up by the passage through it of sufficient current. The collector plate in this gauge is made by application of a conducting coat such as platinum directly to the inner wall of the envelope to which a lead wire may be arranged through the wall of the envelope. Thus the grid and filament may be outgassed by simple application of current, while the envelope and collecting plate may be outgassed either by flaming with a torch or use of a bake oven. The curve shown in Fig. 4·4 is

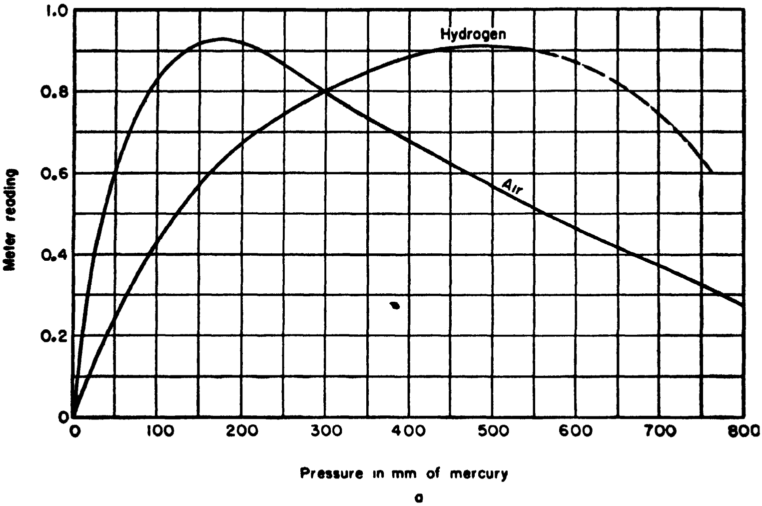
specifically for a gauge of this type, but it will be found to be characteristic of all ionization gauges, the average slope of the curve being an indication of the gauge sensitivity.

In many instances the principle of the ionization gauge may be applied to existing electrodes in an electronic set-up. For instance, in a cathode ray tube sufficient elements are usually present to make possible a measurement of ionization. Calibration may not be known but, if this is of interest, it can be determined quite easily. The circuits associated with ionization gauges are many and varied, although the requirements are quite simple, as was shown in Fig. 4·3. Since the emission current is temperature-limited, variation in the line voltage shows up in the gauge indications and many stabilizing circuits have been worked out to maintain a constant set of conditions in the gauge. At very low pressures the ionization current becomes an exceedingly small fraction of a microampere. This current may be difficult to determine accurately with the average microammeter movement, and d-c amplifier applications have become quite common.

A different approach to the ionization gauge problem is found in one commercial tube.* In this gauge the conventional thermionic tungsten filament and the accelerating grid are replaced by a radioactive source. Response curves are shown in Fig. 4·5. It will be seen that linear relations hold for a useful range of pressures up to roughly one mm Hg and the sensitivity is not great below a few microns. The customary ionization gauge becomes non-linear in this higher range. The explanation probably lies in the fact that the ordinary gauges use electrons accelerated by a hundred or so volts whereas the ionizing radiations of the radioactive source have exceedingly high speeds.

The variation in response of several different gauges to a number of gases [11] is shown in Table II. The value $s = i_p/i_e P$, where i_p is the positive ion current, i_e the electron current, and P is the pressure in microns. Values of s, therefore, are calibration factors for the ionization gauges in different gases. These calibration factors referred to argon are given in the column r and compare well with ionization probabilities $r(P_i)$ obtained at 100 volts.[12]

* National Research Corporation's Alphatron.



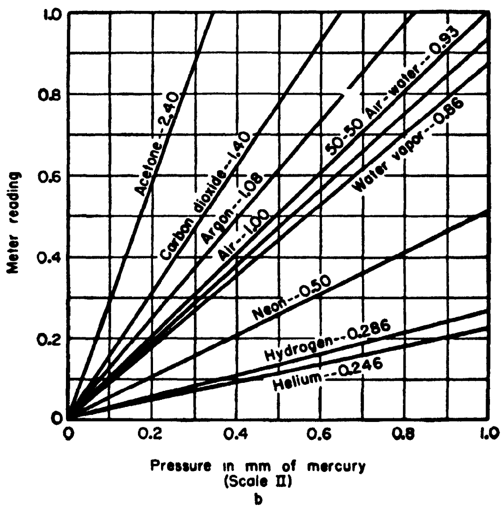


Fig. 4·5 Alphatron Characteristic Curves.

TABLE II

CALIBRATION OF IONIZATION GAUGES WITH ARGON AS A REFERENCE GAS

Gas	FP-62(1)		FP-62(2)		VG-1		Average Value	$r(P_i)$
	$s \times 10^3$	r	$s \times 10^3$	r	$s \times 10^{-3}$	r	r	
He	0.77	0.140	0.70	0.133	2.88	0.127	0.133	0.14
Ne	1.08	0.196	1.08	0.205	4.65	0.206	0.202	0.21
A	5.50	1.0	5.27	1.0	22.6	1.0	1.0	1.00
Kr	8.9	1.62	8.4	1.59	32.9	1.46	1.56	....
Xe	12.0	2.16	13.0	2.46	50.9	2.25	2.29	....
Hg	15.0	2.73	14.5	2.75	72.0	3.18	2.89	1.90
H_2	2.14	0.39	2.10	0.40	8.65	0.38	0.39	0.34
N_2	4.90	0.89	4.40	0.83	18.2	0.81	0.84	0.90

The FP-62 is a General Electric Company gauge.
The VG-1 is a Distillation Products gauge.
From S. Dushman and A. H. Young, *Phys. Rev.*, Vol. 68, p. 278, December 1945.

In the beginning of this chapter it was pointed out that vacuum gauge readings should be accepted with an understanding of their limitations. A good illustration of this fact is found in the application of ionization gauges to systems evacuated by oil diffusion pumps.[13] Two identical gauges connected to the vacuum system through tubulations having tubing speeds * differing by 100 to 1 may show apparent vacuum values of 10 to 1 difference. The explanation in this case probably lies in the fact that the oil vapor entering the gauges and also the molecules broken down by the hot filament and electron bombardment are absorbed by the gauge surfaces. The result is that the gauge acts as a pump. In the case of the low-speed tubing, "replacement gas" from the main vacuum chamber finds it more difficult to get into the gauge with the result that the gauge reading is low. Tubulation speeds differing by 100 to 1 are very likely to be incorporated in typical vacuum systems unless considerable care is exercised.

* For discussion of tubing speeds, see section 9·2.

(*b*) *Thermocouple Gauges.* Cooling of an element at high temperature may be accomplished by allowing the surrounding gas to absorb the energy and carry it to some cooler point in the vicinity. This property of convection is used in several gauges, one of which is the thermocouple gauge. In this gauge a tiny thermocouple is mounted in the vacuum chamber or in a vacuum tube which may be connected to the system. The couple is mounted on an auxiliary heater wire either directly or by means of an insulating glass head. The potential produced by the thermocouple as a result of controlled heating current will depend upon the cooling effect of the gas surrounding the couple. If a sensitive instrument is used to read this potential, the values may be correlated directly with the gas pressure. Customarily, such a gauge is aged by passing through it current 10 per cent or so higher than the operating value. These gauges may be obtained commercially in metal envelopes or in soft or hard glass. They should be calibrated for each type of gas encountered if accurate results are desired. Figure 4·6 shows a typical arrangement of the electrical components. A characteristic curve is given in Fig. 4·7. The sensitivity becomes too small for practical use at about a micron or a little below.

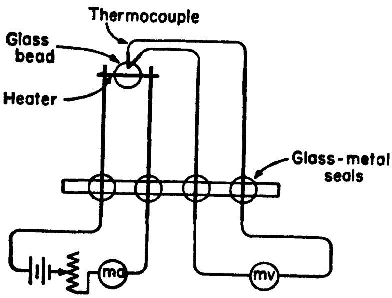


FIG. 4·6 Simple Circuit for Thermocouple Gauge.

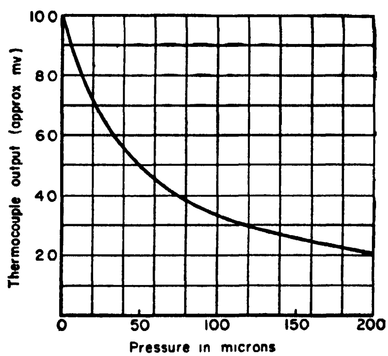


FIG. 4·7 Typical Response Curve for Thermocouple Gauge. (General Electric Company Gauge Tube Catalogue 9651860.)

(*c*) *Pirani Gauge.* The Pirani gauge also makes use of the cooling effect of gas molecules. In this case, the result may be observed as a variation in the resistance of a very fine filament heated by passing a known amount of current through it. This filament is usually incorporated in a bridge circuit as one leg in the bridge and may be calibrated in one of two ways. The out-of-balance indication in the bridge indicator may be calibrated for a fixed current through the gauge, or the current required to maintain balance of the bridge may be obtained as a function of the pressure. This latter method is the more common, since it will cover a wider range of pressures conveniently.

Since the entire bridge and gauge circuit is quite sensitive to temperature fluctuations, it is desirable to insure that the gauge filament and its paired member in the bridge are maintained at the same temperature. This may be accomplished by using as the paired member a second identical gauge sealed off under vacuum and mounted close to the gauge so that temperature fluctuations will cause equal reactions in the bridge. Constant temperature conditions also may be set up by the use of ice water traps or some similar arrangement.

In the construction of a Pirani gauge, greatest sensitivity is achieved when a great change in resistance of the filament can be obtained by a slight variation in temperature; thus, it is desirable to use as long a filament as is convenient with as small a cross-section as is compatible with the fact that a large area for cooling is desired. A good filament may be made of platinum ribbon, which can be obtained a few mils wide by a fraction of a mil thick.

With this gauge, as in the ionization gauge circuits, many elaborate schemes have been worked out to improve the fundamental bridge circuit. Actually, with a little experience very satisfactory results can be obtained with the simple circuit shown in Fig. 4·8. Characteristic curves for one commercial gauge are shown in Fig. 4·9. As in the case of the thermocouple gauge, the Pirani must be calibrated for each gas and the useful range is roughly from 10^{-1} to 10^{-4} mm Hg.

(*d*) *Glow Discharge.* The visible effect produced by ionization of the gas by some device such as a spark coil should not

really be considered as a gauge. However, it is most useful and valuable for the experimenter and should be considered quite seriously as a pressure indicator. Its use in the well-known task of leak hunting will be discussed under that topic. The glow discharge either may be induced by high voltage (a few thousand volts alternating or direct current) applied to electrodes purposely built into a glass tube on the system or it may be initiated by bringing a spark coil near some of the glass tubing in the system. It is possible to judge quite closely the order of magnitude of the vacuum in a system, as it is evacuated down to about one micron. It is probably impossible, though, to describe the appearance of the discharge so that the reader can immediately make an accurate estimate. However, if the system is pumped down from atmospheric pressure a few times and the discharge colors and intensities are observed at various pressures as determined by a suitable gauge, it will be found that not much experience is required to have a fair idea of the condition of the system. A darkened room is desirable for such studies of gas discharges.

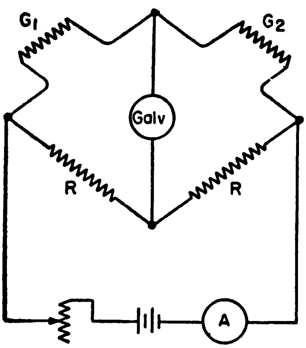


FIG. 4·8 Simple Pirani Gauge Bridge Circuit.

It is also possible at times to judge by such observation whether the system is quite tight, whether it has a leak to air, whether there is some source of contamination giving a virtual leak, or whether the diffusion pumps are trying to operate with insufficient backing from the mechanical pumps. The nature and intensity of the spark used and the diameter of the glass tubing determine, among other variables, at what value of vacuum the discharge may no longer be obtained.

In using spark coils, two general types are available commercially. Typical of them are the two in Fig. 4·10. Type *A* gives what might be termed a hard spark, and great care must be taken not to puncture the glass at points where the wall is thin. The second type is strongly recommended for this work, since it

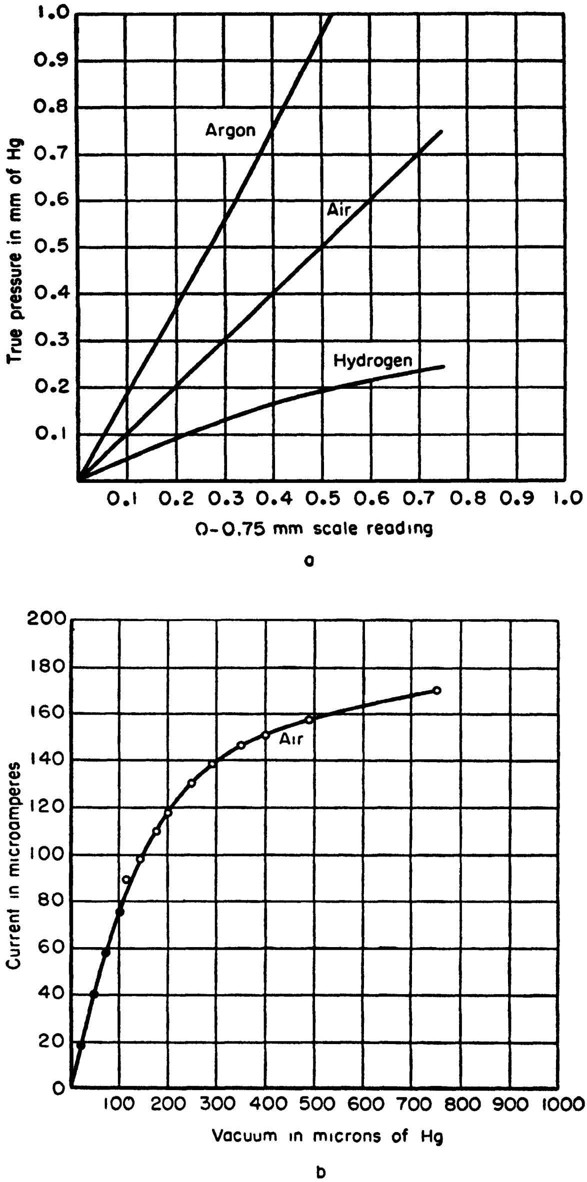
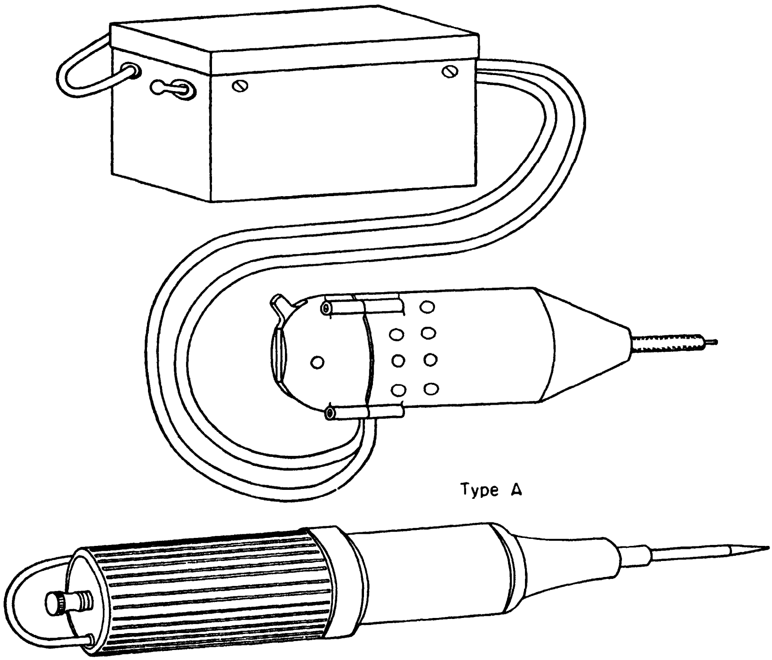


FIG. 4·9 Typical Response Curves for Pirani Gauge. (Distillation Products, Inc., Gauge PG-1A.)

gives a brush discharge at a frequency which gives ionization through the glass much more efficiently. It is practically impossible to rupture glass with this spark coil when obtaining a gas discharge at comparatively low vacuum.



Type B

✓ Fig. 4·10 Spark Coils Used for Creating Glow Discharge.

Fluorescence of the glass walls of the tubing is often encountered, particularly so in systems where there may be a trace of oil or grease. The characteristic color in this case is a pale green or yellow with occasional blue, depending upon the type of glass. It is sometimes surprisingly difficult to differentiate between this fluorescence and a very weak gas discharge.

An ordinary spark coil of the automobile ignition type may be used to produce the spark for this work, and in addition to inducing the so-called "electrodeless" discharge by holding the

spark coil to the glass, a special discharge tube with internal electrodes may be energized by grounding one electrode and connecting the spark coil to the other. In neither case may be recognized the commonly described discharge phenomena containing the well-defined regions such as the Crookes dark space and cathode glow. These are produced by the application of d-c voltages to properly spaced internal electrodes at suitable pressures.

(*e*) *Philips Ionization Manometer.* Not so widely used, although it is an effective gauge, is the Philips ionization manometer. This is an extension of the ionization gauge principle in which magnets are applied externally to the gauge in such a way that the electrons execute spiral paths, and these longer paths give rise to greater ionizing efficiency.

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Chapter 5

VALVES AND CONTROLLED LEAKS

5·1 Glass Valves

As is the case in any plumbing system, of which the vacuum system is merely a special example, it is often desirable to have means for shutting off one part of the system from another. Various types of valves are in common use.

(*a*) *Stopcocks.* Most common in glass systems are the various styles of stopcocks. They are usually lubricated or sealed by means of a special grease, although some models make use of additional mercury seals. These stopcocks are available with a single bore serving as a simple valve or with multiple arrangements of bores, making possible valving of intricate networks of tubing.

Probably the difficulty most often encountered with stopcocks is leakage due to hairlines that develop in the thinned-out grease. This must be watched for constantly and is likely to develop with a single turn of the stopcock or as radical changes in temperature are encountered. If the outer case of the stopcock is held firmly in the hand for a few seconds before applying the turning force, the grease will warm just sufficiently to make turning easier and make less probable the formation of the hairlines.

Another annoyance frequently encountered is the "freezing" of a stopcock due to the grease going dry. As in removing a tight stopper from a bottle, brute force is usually disastrous. A light tapping of the handle at the same time a withdrawing force is applied may sometimes remove the stopcock. If this fails, hot water or a very soft flame played over the body of the valve may give sufficient expansion to make withdrawal possible. A penetrating liquid solvent may also be found useful.

(*b*) *"Dry" Valves.* When absolute cut-off is not required and when extreme cleanliness, that is, freedom from grease, is desir-

able, a ball valve arrangement as shown in Fig. 5·1 is quite satisfactory. The steel ball, which is to be used as the plug, is used with a little grinding compound to form a spherical seat in the glass pedestal. If the grinding is worked through several grades of grinding compound to a fine emery, the valve is extremely efficient. The steel ball may be raised from the pedestal and deposited in the specially provided side tube by means of a magnet externally applied.

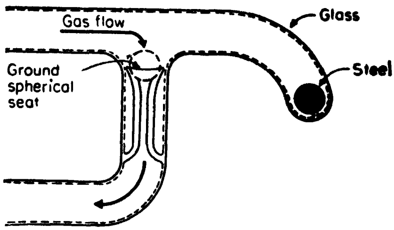


FIG 5·1 Ball Valve.

Another chemically clean valve[1] makes use of a glass plunger closing a silver chloride seat and activated through a flexible glass bellows. Other all-glass devices make use of glass needle valves operated magnetically through glass-coated armatures.[2]

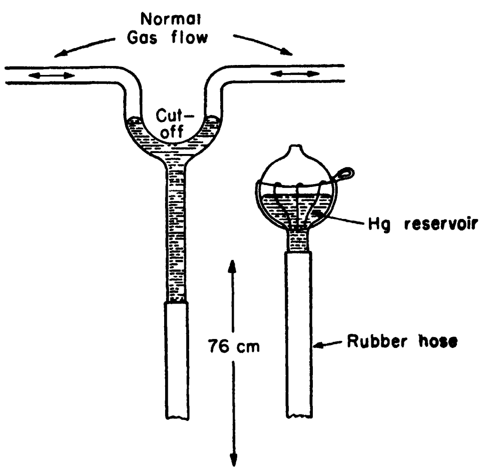


FIG. 5·2 Mercury Cut-off Valve.

(*c*) *Mercury Cut-off.* Complete cut-off by means of a column of mercury which can be raised or lowered has been long known in vacuum practice. Such an arrangement is shown in Fig. 5·2. The device is not usually available as a unit, but

rather the idea is incorporated in the system in the most advantageous way. It will be noted that such a cut-off is inherent in the McLeod gauge discussed in the last chapter. In making provision for raising and lowering the mercury column it should be remembered that atmospheric pressure will raise the mercury about 30 in. and it is wise to design the cut-off so that if the column is accidentally exposed to atmospheric pressure the mercury will not spill over into the vacuum system.

5·2 Metal Valves

(*a*) *Bellows Sealed.* Metal valves available, if we exclude the needle valves used for controlled leaks, conform for the most part to standard valve design. In many cases an ordinary bellows type steam valve may be adapted for use with vacua by sealing certain of its parts with wax or solder or rubber gaskets. Natural or synthetic rubber seats tend to absorb gases which are forced out under the pressure of closing the valve. Silicone rubber apparently exhibits a minimum of this undesirable quality.

It is possible to obtain fairly satisfactory valve action by replacing the sealing portion of the valve stem by a hardened polished piece of steel having a spherical contour which drives into a well-machined edged seat in the body of the valve (Fig. 5·3*a*). This seat, usually of brass, will to a certain extent give under the pressure of the valve in such a way that in some cases nearly complete cut-off is obtained. When such a valve is well made and has not been subjected to grit, which might affect the seat, it is possible to use it between the vacuum system and the pumps with the result that with atmospheric pressure on the one side the molecular diffusion pumps will continue to pump. They should not be used where complete cut-off is essential since they occasionally allow a slight leakage under frequent operation.

(*b*) *Solenoid Valves.* Solenoid drives for vacuum valves are available and make possible remote control. These may be designed so that the magnetic field is operative with the valve open or conversely with it closed. Such valves must many times be water-cooled, and the strong fields required usually cannot be tolerated near electron beam equipment. One such valve has been designed which is energized only during the opening or

closing operation, thus eliminating both the above-mentioned disadvantages. The general idea of this valve is illustrated in Fig. 5·3*b*. Two electromagnetic coils are used spaced so that each one tends to pull the plunger in its own direction. Thus, by energizing one or the other of the magnets, the valve can be opened or closed. Detents are provided to hold the plunger in place at either position. For very large valves the use of ex-

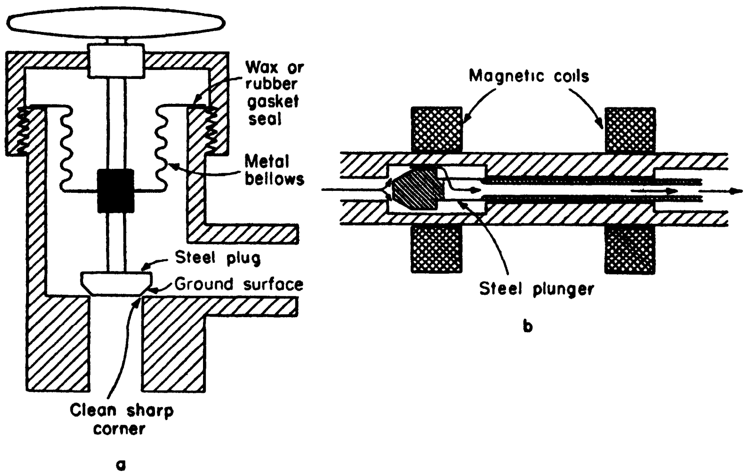


Fig. 5·3 Vacuum Valve Designs.

cessively large coils may be obviated by the use of smaller "bleeder" valves which open first.

(*c*) *Air Bleeders.* In many vacuum systems where it is desirable to open and close the system frequently there is the problem of admitting air by some convenient method. In glass systems a small side tube with a drawn-out tip is convenient. The tip is easily broken off when desired and readily sealed again for closing the system.

An extremely satisfactory solution for metal systems is found in the very well-known and economical tire valve. Several variations of this application have been described.[3] Perhaps the simplest is illustrated in Fig. 5·4. An extension is attached, either by spot welding or soldering to that portion of the valve insert which normally is inside the tire. The insert, together with

the valve stem, is incorporated in the wall of the vacuum system so that this handle is convenient, and when it is desirable to admit air to the system it is merely necessary to hold this

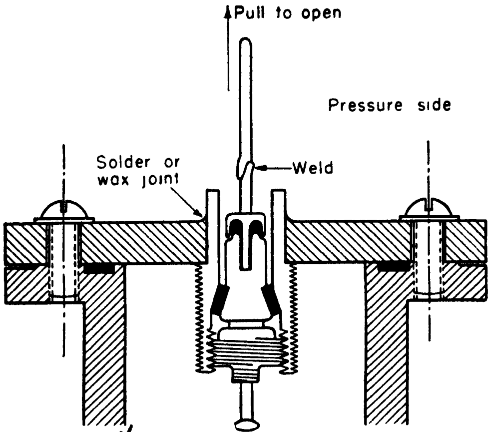


FIG. 5·4 Bleeder Made of Tire Valve.

valve open. Since some spring loading is available, the scheme may also be used by remote control means in any application where a volume at one gas pressure is to be opened to a volume at another gas pressure. In addition to the economy and con-

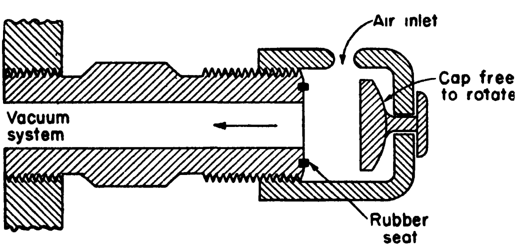


FIG. 5·5 Bleeder Design.

venience of this device, it satisfies good vacuum practice in that an extreme minimum of rubber is exposed to the vacuum.

Another design of such an air inlet is shown in Fig. 5·5. Here a flat cap is forced down on a rubber washer. Grabbing of the

cap by the rubber when the screw cover is turned is eliminated by having the cap rotatable with respect to the cover. The shaft is kept small to minimize frictional drag.

5·3 Controlled Leaks

In many instances it is desirable to maintain a constant flow of gas through the vacuum system for some purpose; for instance, it may be desirable to carry on work at a pressure of a few microns of some specific gas. To accomplish this by ad-

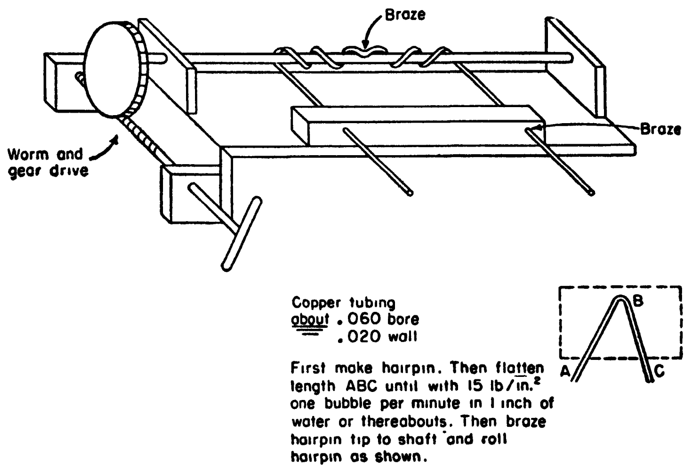


FIG. 5·6 Variable Leak of Flattened Copper Tubing.

mitting the gas to a previously exhausted system and pumping it down to, say, 10 microns may result in considerable contamination of the gas by continuously evolved residual gases as well as by some action of the experiment itself. Much better results may be accomplished if an arrangement is possible whereby the desired gas is admitted to the system through a slow leak and caused by the pumps to flow throughout the region. In certain X-ray tubes the presence of gas at a reduced pressure is necessary for operation, and in this case a controlled leak is provided as a satisfactory solution. Other instances of a similar nature may present themselves, and all the following mechanisms have been tried with success.

(*a*) *Glass Capillaries.* One of the oldest known slow leaks consists of an extremely fine capillary of glass. A piece of thick-walled glass tubing is heated uniformly with a torch near its

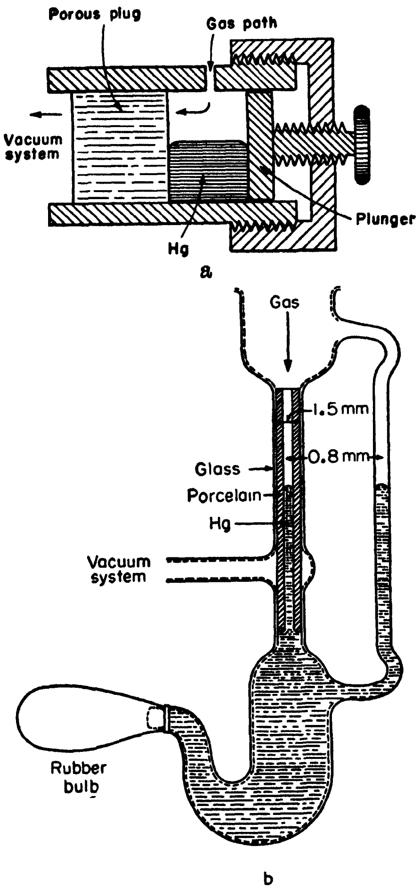


FIG. 5·7 Controlled Leaks Through Porous Material.

center until the glass commences to thicken and sag. If, while the tube is in this condition, the two ends are suddenly pulled far apart, possibly to the extent of several feet, a very small capillary tubing results. If this tubing is attached to the vacuum system, the attenuation of the passage of gas through the capil-

lary will result in an effective slow leak. If the leakage is too slow, its speed may be increased by breaking off lengths of the tubing until the desired value is reached. If the rate of leakage is too fast, then, of course, a finer capillary must be drawn.

(*b*) *Rolled Metal Tubes.* If a length of copper tubing is carefully cleaned, bent to a hairpin shape, and carefully flattened, it may be converted into a controlled leak by making rigid to a frame the two ends of the hairpin while the bend is carried over

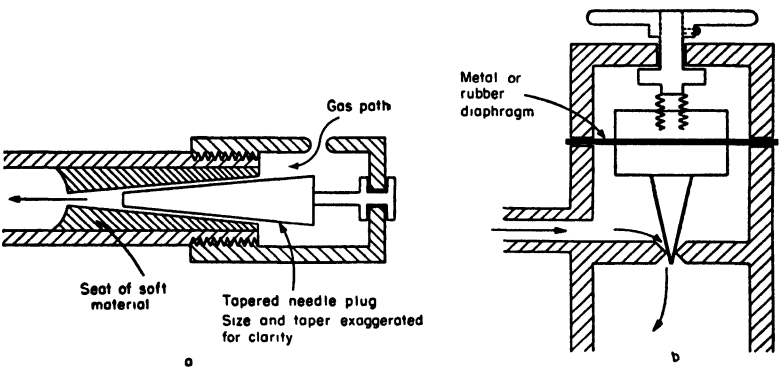


Fig. 5·8 Needle Valve Types.

a cylinder and is bent around in such a way as to continue the original flattening.* The bend in the hairpin is attached to the cylinder, which may be driven by a worm gear or a controlled lever in order to hold constant any position which has been found to give a desirable value. Figure 5·6 shows such an arrangement. A simpler form makes use of a vise-like clamp to control the flattened tubing.

(*c*) *Porous Plugs—Mercury Limited.* Many arrangements have been described wherein some porous material is arranged so that its length or the amount of its cross-section available for gas leakage is controlled by covering it with a layer of mercury. A number of porous materials are satisfactory for this purpose, one of the most common being unglazed porcelain. Two such ar-

* This device was developed in connection with work under the direction of Professor A. O. C. Nier of the University of Minnesota, Minneapolis, Minn.

rangements are shown in Fig. 5·7, two variations of the 5·7*b* being described in the literature.[4, 5]

(*d*) *Needle Valves.* The applications of needle valves to this problem have been many and varied. In general, a very fine screw drive controls the position of the needle, which may be a

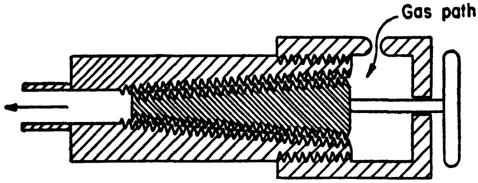


FIG. 5·9 Tapered Screw-Controlled Leak.

steel-tapered plug several inches long and a few mils in diameter, and which may be fitted into a hole in a softer material such as lead or tin. A workable model is shown in Fig. 5·8*a*. In Fig. 5·8*b* is shown a model[6] which makes possible controlled leakage between two regions, adjustment being made from outside the system.

(*e*) *Tapered Screws.* In another arrangement, Fig. 5·9, similar to the needle valve, the path length is increased immeasurably by the use of a tapered screw which engages in a hole having a matched tapered thread. This device is effective enough but requires considerable skill in manufacture.

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Chapter 6

DEMOUNTABLE JOINTS

6·1 General Discussion

It should be obvious by now that work with a vacuum system really is a glorified sort of plumbing. Joints, pipes, valves, gaskets, and other terms common to ordinary plumbing are found in this art. In carrying out experimental work in electronics in a vacuum system often much time can be saved if the experiment can be set up in a demountable chamber. By using various component sections, some of which may at other times be used with other experiments, a vacuum-tight housing is assembled which will satisfy the requirements of the experiments. Such requirements may dictate the use of glass, metal, or both.

Peculiar shapes may be required to make provision for specific electron paths or to accommodate the mechanisms with which the electron beams are to be studied. In this case glass or metal tubes may be made up from components similar to the fittings used in ordinary plumbing. For instance, elbows, T sections, extensions, etc., as desired, may be assembled by means of wax or rubber gaskets. A complete cathode ray tube may be assembled, for example, in such a way that various electron guns, deflection plates, focus means, or fluorescent screens may be interchanged and replaced by others without the necessity of fabricating a complete tube for each test. Figure 6·1*a* shows such an arrangement in glass ready to be waxed together; Fig. 6·1*b* shows the same arrangement of parts in a standard sealed-off tube. Of course, complete outgassing is not possible under such conditions, but it is not necessary since the system will be pumped continuously during the experiment. A number of types of joints used in assembling demountable equipment are described here.

6·2 Wax and Putty

Under waxes are classed the various compounds which, upon cooling, are quite rigid, but which, when warmed, may be easily worked in the preparation of a vacuum-tight joint. Ordinary

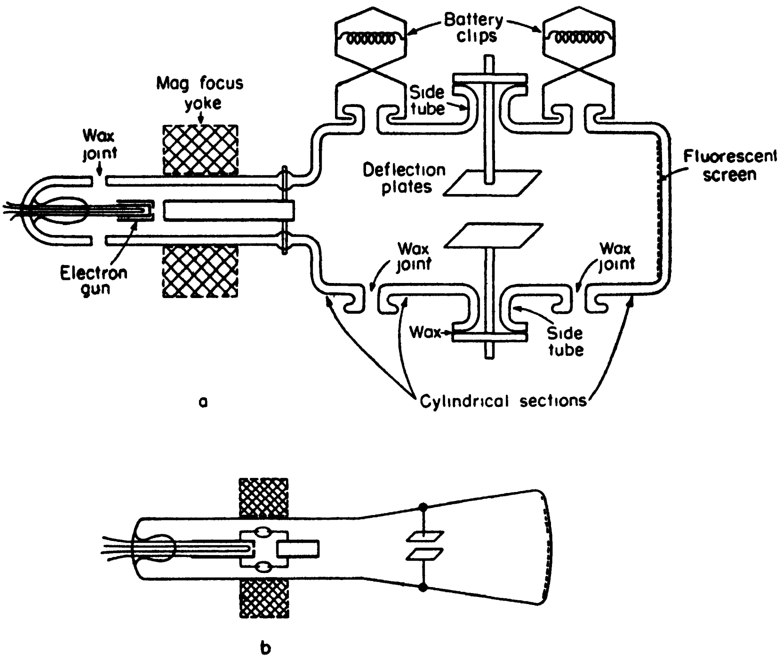


FIG 6 1 Typical Demountable Vacuum Chamber for Electron Beam Work

sealing wax is very much used, although its vapor pressure makes it undesirable where extremely high vacua are to be attempted This wax becomes fairly brittle when cool and should not be depended upon for its mechanical strength It is somewhat soluble in alcohol and when softened in that way may be applied with a brush It also may be applied by a flame or by putting the normal wax to a surface hot enough to melt it, or it may be transferred to the work by means of a rod which is kept sufficiently warm

Waxes having better properties than sealing wax are De Khotinski and Sealstix.* They have a shellac base and may, although with some difficulty, be dissolved with alcohol, chloroform, ether, or caustic solutions when it is desired to remove them from the work. Care must be taken not to overheat them since they decompose quickly under that condition and become quite difficult to remove. Sealstix works at about 140°C.

Picein is a black wax with properties somewhat similar to the De Khotinski and sealing wax, except that it has a greater softening range and can be worked at somewhat lower temperatures. It can be dissolved by turpentine.

A very convenient series of waxes is furnished under the trade name Apiezon.† (See Table I.) Apiezon wax W-40 is a black

TABLE I

APIEZON WAXES

Product	Use	Vapor Pressure, in mm Hg	Max. Temp.	Temp. for Application
Soft Wax Q (Sealing compound)	Unground joints	10^{-4} at room temp.	30°C	60°C
Medium Soft Wax W-40 (Sealing compound)	Unground joints	10^{-3} at 180°C	30°C	40° to 50°C
Medium Hard Wax W-100 (Sealing compound)	Permanent joints	10^{-3} at 180°C	50°C	80°C
Hard Wax W (Sealing compound)	Permanent joints	10^{-3} at 180°C 10^{-6} at room temp.	80°C	100°C

From the suppliers' literature.

material having low vapor pressure and a consistency about like that of tar. When warmed by working with the fingers, it may be molded to some extent. Sealing wax W comes in sticks convenient for application and cannot be worked by the hand. It

* Central Scientific Co., Chicago, Ill.

† James Biddle Co., Philadelphia, Pa.

may be applied with a torch or hot rod or it may be taken up by the work itself when it is heated. This material has a tendency to be brittle when cold, and care should be taken that its mechanical strength is not depended upon. Wax W-100 has characteristics between those of W and W-40.

There is also available a vacuum putty which is quite useful. This material (Apiezon soft wax Q) has the property of being quite easily worked with the hands and of not flowing as easily as a grease. For example, it may be used around the base of a bell jar in much the same way that ordinary putty would be used, and it has a vapor pressure sufficiently low to make its use feasible (see Table I). All these waxes are soluble in a number of solvents, including carbon tetrachloride, ethylene dichloride, and ether.

Although it probably should not be classed as a wax, silver chloride has certain properties which call for its being included as such. It adheres well to glass and metal and is useful for work where temperatures higher than those permissible with ordinary waxes are encountered. It melts at about 450°C and is soluble in sodium thiosulphate.

In the assembly of any joints of a demountable system the use of a minimum amount of wax should be the rule. One method which has proved satisfactory for aiding in the assembly of a system of several components is as follows. The openings of the various glass articles to be joined are flared outward to the same diameter and provided with a bead (Fig. 6·1). These surfaces are then ground so that flats are presented to each other when they are joined. In assembling, these flanges are warmed with a torch until a wax such as Apiezon wax W will adhere. With each part properly waxed, the flanges are brought together, and several spring type battery clips of suitable size are clipped over the beads of the flanges. Upon the application of sufficient heat from a torch the wax will soften and the flanges will be pulled together by means of the battery clips, which may be left on to give mechanical strength if it seems necessary. Whenever possible, regardless of the shape of the joint structure, it is desirable to obtain as good a mechanical fit as possible so that the wax need only perform its task of making the joint vacuum-tight.

6·3 Grease

A large number of greases are now available for vacuum work. They vary in their consistency and vapor pressures. A light and

TABLE II

VACUUM GREASE CHARACTERISTICS

Product	Use	Vapor Pressure, in mm Hg	Max. Temp.
Apiezon Grease L	Closely fitting ground joints (not for stopcocks)	10^{-3} at 300°C; unmeasurable at room temp.	30°C
Apiezon Grease M	Places where a grease of moderately low vapor pressure is required	10^{-3} at 200°C; 10^{-6} at room temp.	30°C
Apiezon Grease N	Stopcocks; also places exposed to heat, and where a grease of higher viscosity is desired	Unmeasurable at room temp.	30°C
Celvacene, light	Stopcocks, ground joints, etc., in high temperature application	Less than 10^{-6}	Melting point above 90° C
Celvacene, heavy	Rubber gaskets, rubber to metal joints, etc.; high temperature	Less than 10^{-6}	Melting point above 130°C
Vacuseal, light	Stopcocks, ground joints		Melts above 50°C
Vacuseal, heavy	Stopcocks, ground joints		Melts above 60°C
Silicone	Stopcocks, ground joints		Workable from −40°C to 200°C

From the suppliers' literature.

a heavy bodied grease are available under the name Vacuseal.* Others are the line of Apiezon † stopcock greases and two greases of different consistency under the trade name Celvacene.‡ In

* Central Scientific Co., Chicago, Ill.
† James Biddell Co., Philadelphia, Pa.
‡ Distillation Products Co., Rochester, N. Y.

addition, a grease of Silicone * is also available. These greases may be used in joints where the mechanical fit is quite good, and where it is necessary to have a sliding fit or a joint which is to be opened frequently such as tapered ground joints and stopcocks. Great care must be taken in their use to insure against their becoming too warm, and in the stopcock applications extreme cold must also be avoided or it will be impossible to operate the moving parts. (The Silicone products have improved thermal characteristics.) These greases should be renewed frequently in most applications since with most joints that are tapered the effect of the external pressure will be gradually to squeeze the wax from the ends of the joint. Table II on preceding page lists characteristics of some of the greases.

By no means all the known waxes and greases have been described here since not all are equally pertinent to the work. More complete treatments are available in the literature,[1, 2, 3] and it is recommended that they be referred to if the materials described above are not adaptable to the specific case.

6·4 Sealing Paints

The use of sealing paints such as glyptal,† lacquer, or shellac plays an important part in vacuum work.

There are times when neither wax nor grease has quite the properties desired. Occasionally the seams of a metal structure may have a number of pinholes, and it is undesirable or impossible to refabricate the part, or a vacuum wall may be found to be porous. Small leaks may be found in locations such as in glass tube stems where the application of wax is difficult, or leaks may be encountered which are so large that grease will not hold against the vacuum. In these and many other cases the use of a liquid sealing agent is invaluable.

Often painting the complete system is a last-resort method of obtaining a vacuum. Incidentally, observation of a gauge while such painting is being done often is a good way to detect leaks. In such application it is sometimes advantageous to use the colored forms of the paint to aid in locating the region treated.

* Dow Corning Corp., Midland, Mich.

† General Electric Company, Schenectady, N. Y.

The consistency of glyptal is quite easily controlled by its thinner. Large leaks may be closed with it in the thickened state, and at the other extreme it may be thinned sufficiently to be used with an atomizer in closing extremely fine holes or porous materials.

One of the characteristics of these paints is that they are drawn into the leaking orifice, and as the volatile solvent is removed by the vacuum the residue does a satisfactory job of plugging. This very trait may prove to be a disadvantage, however, if plans call for more permanent repair of the region. These plugs carbonize as the glass is reworked and may themselves make it difficult to repair the part. If the structure is metal there may be difficulty in cleaning the residue away enough to do a good solder or weld job.

6·5 Mercury Seals

In a few instances mercury may be used to obtain a good vacuum seal which may be opened or closed quite easily. If a sufficiently good mechanical joint is available, the mercury will not

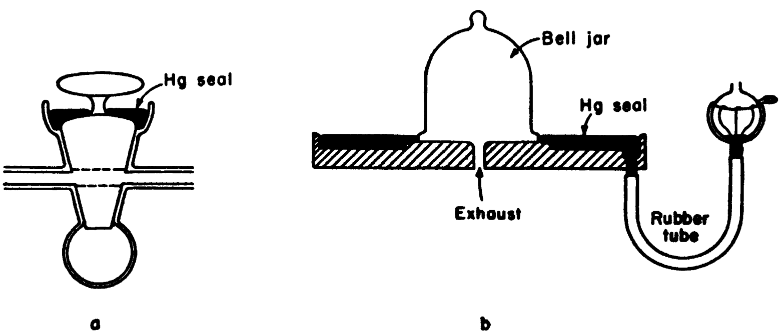


FIG. 6·2 Methods of Mercury Sealing.

be pulled into the vacuum system. Occasionally a very light coating of grease is applied to a ground joint to insure that this does not happen. Some stopcocks are arranged to accommodate a mercury seal.

Another useful application is sealing a bell jar to its base plate. In this case the flat upon which the bell jar rests is provided

with a rim into which the mercury can be poured after the bell jar is properly seated. Provisions for draining away the mercury are easily made. One scheme makes use of a mercury reservoir connected to the trough around the bell jar by means of a flexible hose. Raising or lowering of the reservoir will flow the mercury around the bell jar or drain it from the trough. Figure 6·2 shows the two applications described here and may suggest others. Good ventilation should always be provided where mercury is exposed since severe mercury poisoning may result from inhaling the vapor.

6·6 Gaskets

The assembly of metal components of a demountable vacuum system using rubber gaskets has become more and more prevalent. Although it is recognized that the exposure of large areas

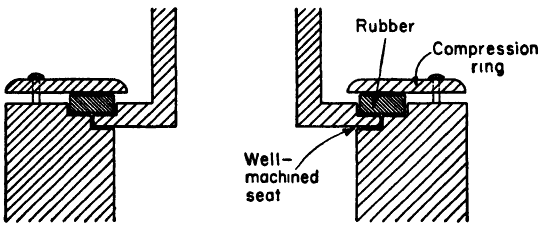


FIG. 6·3 Gasket Seal of Good Design.

of rubber in a vacuum is deleterious, it is possible by the proper design of the joints to use rubber gaskets without appreciable effect on the ultimate vacuum reached. The general principle followed is to build the joint so that the two elements to be joined make a union which is mechanically good in itself, and which leaves only a very narrow machined crack to be stopped by the gasket. The gasket then is applied under pressure in such a way that it cannot spread and is forced to perform the sealing action. Figure 6·3 shows the basic principle of this type joint. Some installations may require variations to fit the conditions encountered. When possible, the machining of the two members should provide a very narrow and long leak path.

In some cases such a joint cannot be used for one reason or

another, and if the pumps are sufficiently fast and the rubber is of good grade considerable area of exposure can be accommodated while still achieving a good vacuum. Figure 6·4 shows a number of useful joint designs. Those shown in *a* and *b* are useful for joining metal to glass tubing.

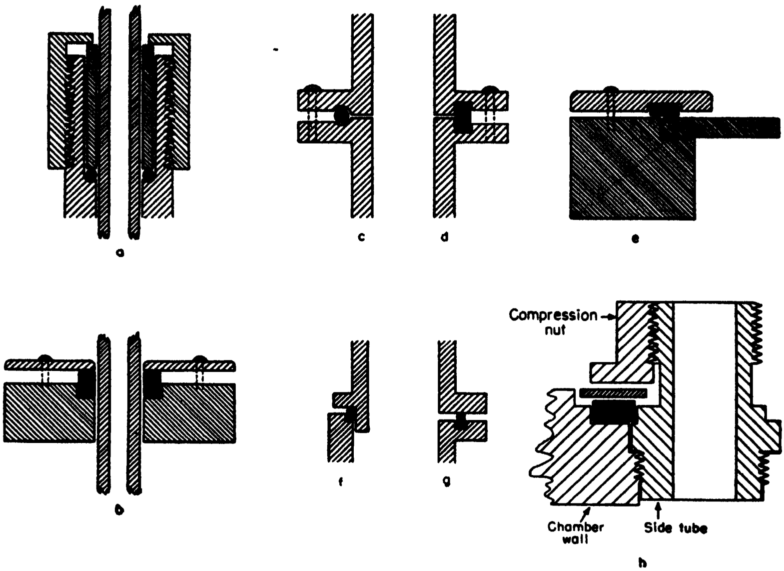


Fig. 6·4 Gasket Joint Designs.

Although many rubber substitutes have become available, considerable care should be taken in their application as most of them will take a permanent set upon prolonged application of pressure. This set may not be undesirable in some instances, and sometimes the ability of these synthetic materials to resist oil and grease may make them particularly desirable. Some applications of metal gaskets have been made. Here some metal which will flow readily under pressure, such as aluminum or lead, is used. Usually in these cases a ring of metal will flow enough to give a good seal. When once broken, these joints usually must be remade with a new gasket, since the original one is completely deformed.

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Chapter 7

CONTROLS AND GADGETS

7·1 Magnetic Fields

It has been pointed out that in assembling an apparatus for conducting an electronic experiment in vacuum it is often necessary to provide suitable means of controlling mechanical or electrical components inside the vacuum. Many ingenious schemes have been evolved; some have become common knowledge but others are not so well known.

Magnetic forces may be used to obtain mechanical motions for remote control in a number of ways. Sometimes the magnetic fields are formed and used within the system; at other times the force may be exerted through the walls. In the case of internal controls, mechanical motions may be actuated by means of electromagnets energized by current carried by leads through the chamber wall. In other instances, magnets may be arranged just outside the walls, but close to them in such a way that the magnetic fields will react with iron armatures or other magnets just inside the container. Obviously the use of any but nonmagnetic materials in the chamber wall complicates the situation.

With devices of this kind, rotation of an object in the vacuum may be accomplished at almost any desired speed. The part to be rotated is mounted in the proper bearings and connected to an armature arranged so that its field is provided by coils or magnets outside the chamber. Such arrangements are shown in Fig. 7·1.

Surfaces of barium or silver may be evaporated or plated upon bearings to avoid "seizing" during vacuum operation. Molybdenite is also useful as a dry lubricant for vacuum mechanisms.

If a linear motion is required, it may be obtained with the scheme shown in Fig. 7·2, where movement of the magnet along the outside of the chamber is followed by the internal armature

attached to the member to be controlled. Rotation and linear motion may be combined in such a device. The solenoid valve

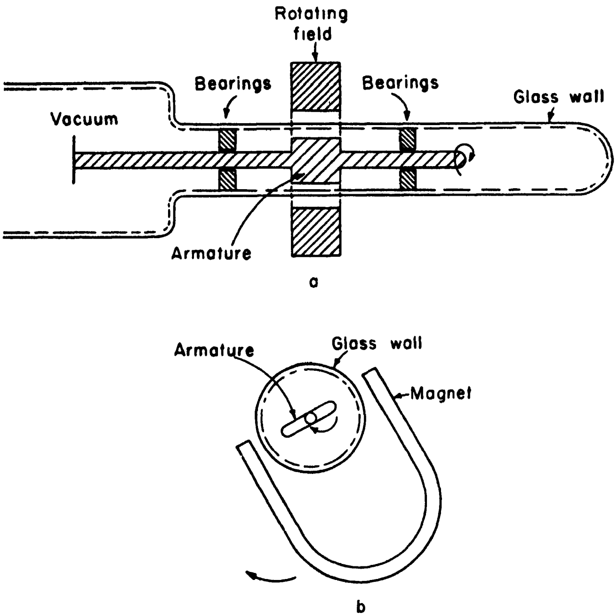


FIG. 7·1 Magnetic Means of Obtaining Rotary Motion in Vacuum System.

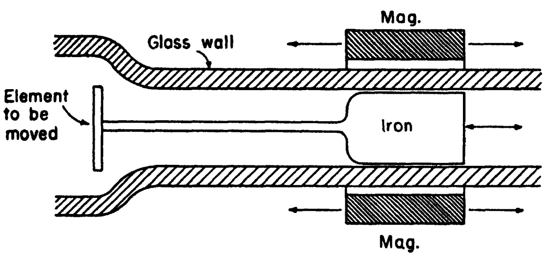


FIG. 7·2 Linear and Rotary Motion by Magnetic Control.

described in Chapter 5 is a practical application of this idea. In that case the iron plunger in the valve is moved back and forth in its housing by alternate application of spaced magnetic fields outside the vacuum walls.

The effect of magnetic fields upon charged particles many times will limit or prevent the use of this type of control in electronic systems. The use of magnetic fields to achieve control of charged particles will be discussed in a later chapter.

7·2 Electric Current

Electric current can be used in a variety of ways for remote-control purposes. Some of these ways may be to perform mechanical motions; others will merely fulfill the requirements of providing the experiment with the necessary currents and potentials to affect the electron or ion beam. The effect of the electric fields on charged particles will be discussed later as will the subject of electrically heated thermionic cathodes.

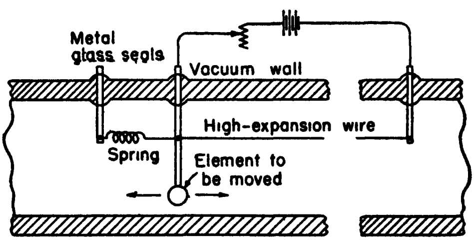


FIG. 7·3 Motion in Vacuum Through Controlled Linear Expansion.

One method of obtaining mechanical motion involves the passage of current through a device for the purpose of changing its temperature and thus its physical configuration. For instance, a bimetallic strip may be heated either by an auxiliary heater or by passage of the current through its elements to obtain a very pronounced mechanical force.

The expansion of materials under the application of heat may be used in many other ways, for instance, the extension of a long filament of wire may be used to operate a shutter (Fig. 7·3) or some similar mechanism. Many times the mechanical device may be arranged so that a motion or series of motions may be set off by the burning out of a link as shown in Fig. 7·4. Such motions may be driven by gravity, by spring force, or by some other such means. This idea is somewhat similar to that used

in holding open fire doors which close of their own accord when the fuse link reaches the critical temperature and breaks the restraining force.

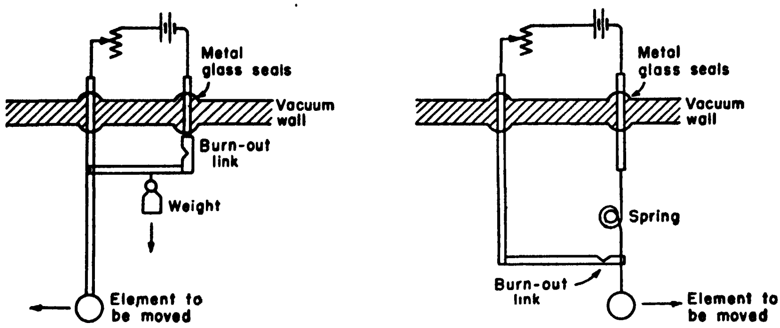


FIG. 7·4 Remote Control by Triggered Release.

7·3 Materials Having Elastic Properties

A number of arrangements using rubber washers or tubing may be used to obtain rotary or sliding motion. Many variations of the technique using rubber washers are described in the literature, [1,2] and simple forms of the device are shown in Fig. 7·5.

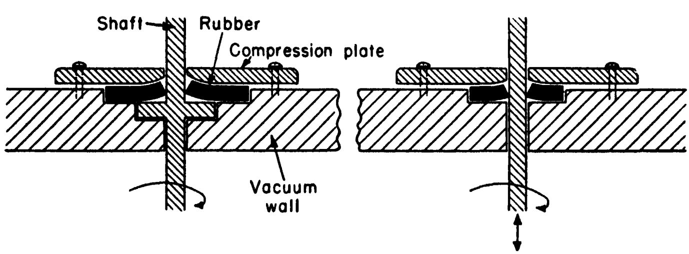


FIG. 7·5 Vacuum System Motions Through Rubber Seals.

The shaft by means of which the motion is carried passes through a rubber gasket, the clearance hole in which is smaller than the diameter of the rod. It is arranged that the resulting deformation of the rubber is such that the high-pressure side of the seal tends to force the rubber against the shaft; and, although it is not essen-

tial, a small amount of vacuum grease insures free sliding or rotary motion with a vacuum-tight joint.

Rubber tubing may be used to obtain either unlimited rotary motion or limited translation and rotation. The scheme giving rise to unlimited rotation makes use of a crank, the handle of

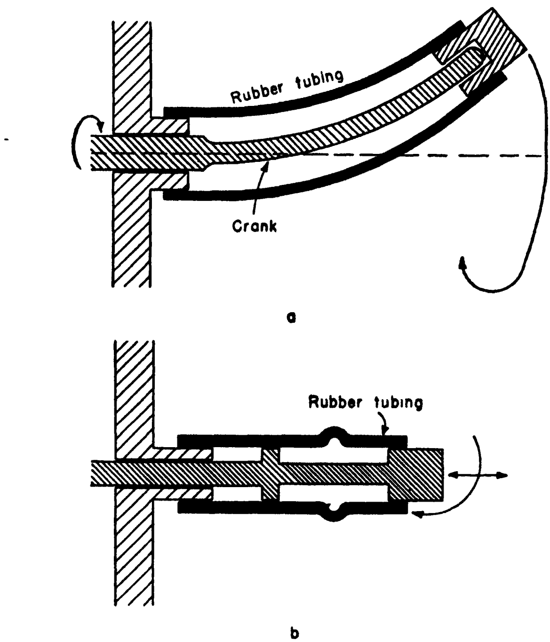


Fig. 7·6 Remote Control Motions Using Rubber Tubing.

which is enclosed in rubber tubing in such a way that the rubber flexes on the crank handle. The resulting rotary motion may be converted to other motions by means of gears, screws, or cams. Figure 7·6*a* shows a typical arrangement of this kind, and Fig. 7·6*b* shows a method of obtaining translation or rotation limited by the elasticity of the rubber.

In some of the forms of antivibration mountings the rubber is well bonded to the metal. When the amount of exposed rubber can be tolerated, some of these mountings may be used to introduce limited motions into the system.

7·4 Metal Bellows

The possibility of deforming thin sections of metal as in bellows and diaphragms has been exploited for remote-control purposes. Metal bellows may be used in connection with a crank mechanism in a way similar to that just described using rubber tubing. In such case the rubber is replaced by the bellows. Because of the peculiar deformation of the bellows which results in this application, great care must be used in designing the crank. In all metal bellows applications the design should incorporate some restraint so that the bellows will not be deformed by excess contraction or expansion due either to pressure or mechanical operation. With proper design such an arrangement will operate through many thousand turns before failure. On the other hand, it is not uncommon to have the bellows fail within twenty or thirty revolutions because of the twisting imposed by improper design. Well-designed cranks of this type are available commercially.*

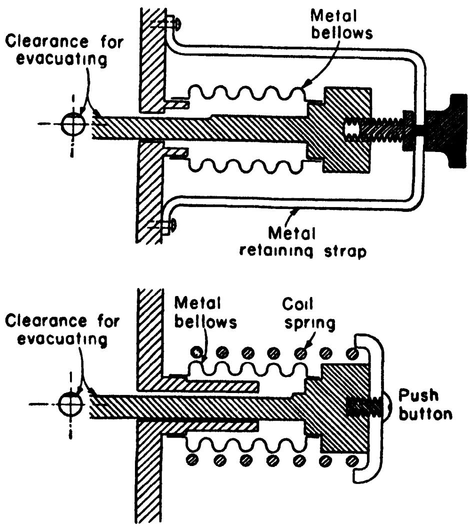


FIG. 7·7 Linear Motion Using Metal Bellows.

* Kearfott Engineering Company, New York City. National Research Corp., Boston, Mass.

Linear motion is more easily accomplished with metal bellows, which are extremely reliable, provided the bellows specifications are not exceeded. Typical arrangements for providing this motion are shown in Fig. 7·7.

The needle valve shown in Fig. 5·8*b* is an application of the principle of diaphragm deflection. The diaphragm may be of rubber or thin metal. The base of the common oiling can is a good example of the metal diaphragm principle. It is often provided with concentric ridge and groove-like deformations for greater flexibility.

7·5 Physical Manipulation

In certain cases where the tube is to be sealed off after evacuation, it is possible to accomplish certain mechanical functions inside the tube by physical manipulation at that time. For instance, counterweights may be applied to shutters, apertures, and similar devices in such a way that they can be made to function by tilting the entire tube. In case the device is being exhausted through a length of rubber tubing, this same scheme is possible during the pumping process.

REFERENCES

1. R. R. Wilson, *Rev. Sci. Instruments*, Vol. 12, p. 91, February 1941.
2. L. Marton, *J. Applied Phys.*, Vol. 16, p 136, March 1945.

Chapter 8

GLASS-BLOWING FUNDAMENTALS

8·1 Equipment

Only a few of the fundamentals of glass blowing are required to make possible a working technique which is adequate for most of the minor repairs and assembly problems around a vacuum system. The building of glass pumps and traps and large-scale apparatus requiring metal-glass seals and heavy fires may well be left to the professional glass blower. Most of these items may be purchased commercially. The ability to make simple seals, however, is desirable and is a skill quickly attained.

The glasses most commonly used in laboratory work are Pyrex and Nonex (boro silicate hard glasses) and lead glass and lime glass (soft glasses). The latter two are more commonly used in high-production commercial work, as lamps, radio tubes, and sign tubing, whereas Pyrex is the most satisfactory glass for laboratory work since it is relatively shock-proof to reasonable temperature changes, anneals quite easily, and softens and works at reasonable temperatures.

Many seals of glass to other materials are possible provided the glass matches the material in certain physical characteristics.[1] For such purposes many special glasses have been developed. A list of materials commonly sealed to glass, together with the code numbers of the glasses used, is shown in Table I. The physical characteristics of the glasses are shown in Table II. When such seals are formed using glasses quite different from the glass of the main vacuum system, it is customary to use a "graded seal" consisting of a number of sections of glass of differing expansion coefficient arranged so that there is no excessive difference in expansion coefficient in the completed joint.

For working Nonex or other glasses containing lead, care is required to keep the work in an oxidizing atmosphere to avoid

blackening due to reduction of the lead salts. For this reason oxygen-hydrogen flames are usually not used with these glasses.

TABLE I

METAL-GLASS COMBINATIONS

Material	Approx. Expansion Coefficient per Degree Centigrade	Sealing Glasses (Corning Code Numbers)
Porcelain, unglazed	40×10^{-7}	772 (Nonex), 774 (Pyrex)
Tungsten	50×10^{-7}	705, 706, 3320, 7991, 772, 775 (to 772 better with 705 as wetting glass)
Tungsten (less than 0.020 in. diam.)	50×10^{-7}	774
Fernico, Kovar	50×10^{-7}	704, 705, 7052, 706, 752
Molybdenum	50×10^{-7}	704, 705, 7052, 706
Molybdenum (less than 0.020 in. diam.)	50×10^{-7}	772, 774
Platinum	90×10^{-7}	001, 008, 012, 756
Palladium	110×10^{-7}	001, 008, 012, 756
Platinum (foil ribbon)	90×10^{-7}	772, 774
Iron (SAE-1010)	140×10^{-7}	1990, 1991
Stainless steel (Sealmet No. 28, Alleg No. 55 controlled)	135×10^{-7}	024
Sylvania No. 4, Driver-Harris No. 14		001, 008, 012
Dumet (up to 0.020 in. diam.)		001, 008, 012
Copper	170×10^{-7}	Seals to most glasses with feather edge (Housekeeper seals)

This table shows those materials and glasses which have been satisfactorily sealed together in electronic work. More detailed information may be obtained from the Technical Service Dept., Corning Glass Works, Corning, N. Y.

Pyrex does not require such particular flame adjustment and is by far the most satisfactory glass, particularly for a beginner. For that reason the following discussion will assume the use of that glass unless otherwise stated.

TABLE II

CHARACTERISTICS OF SOME CORNING GLASSES

Code	Laboratory No.	Notes	Expansion Coefficient, per Degree Centigrade	Softness Point, °C	Annealing Point, °C	Strain Point, °C	Approx. Working Point, °C
001	G-1	Clear potash soda lead—lamp stems—some conductors	91×10^{-7}	626	428	397	970
008	G-8	Clear lime—bulbs	92×10^{-7}	696	510	475	1000
012	G-12	Clear potash soda lead—radio stems—non-conducting	89×10^{-7}	630	433	400	975
024	G-125-BB	Chrome steel sealing glass (Sealmet No. 28, Alleg No. 55)	96×10^{-7}	607	425	397	
1990	G-189-IY	Low-loss iron sealing	127×10^{-7}	496	359	334	
1991	G-184-ET	Iron sealing	128×10^{-7}	539	393	366	
3320	G-371-BN	Heat-resistant Canary, Nonex-Pyrex intermediate—tungsten sealing	40×10^{-7}	780	535	497	
5420		Heat-resistant blue tubing—Nonex-Pyrex intermediate	35×10^{-7}	784	540	499	
704	G-705-Ba	Special sealing	47×10^{-7}	702	484	450	1080
705	G-705-AJ	Special sealing	46×10^{-7}	703	496	461	
7052	G-705-FN	Special sealing	46×10^{-7}	708	480	442	1115
706	G-705-AO	Special sealing	50×10^{-7}	690	495	463	
752	G-750-AJ	Special sealing	61×10^{-7}	747	566	530	
756	G-750-AL	Special sealing	86×10^{-7}	702	536	503	
772	G-702-P	Heat-resistant clear Nonex	36×10^{-7}	755	518	484	1110
774	G-726-MX	Heat-resistant clear chemical Pyrex	32×10^{-7}	820	553	510	1220
775	G-705-R	Clear sealing	40×10^{-7}	704	467	431	
7991	G-704-EO	Tungsten sealing	41×10^{-7}	800	565	529	1175

This table includes those glasses most commonly required in experimental electronics. A more detailed list of Corning glasses may be obtained from the Technical Service Dept., Corning Glass Works, Corning, N. Y.

Usually the work around a vacuum system may be carried out by means of a hand torch which may use gas and oxygen or hydrogen and oxygen. Most convenient is the type having controls on the torch for adjusting the flame, since everything from a needle point to a large brush flame may be used in a simple

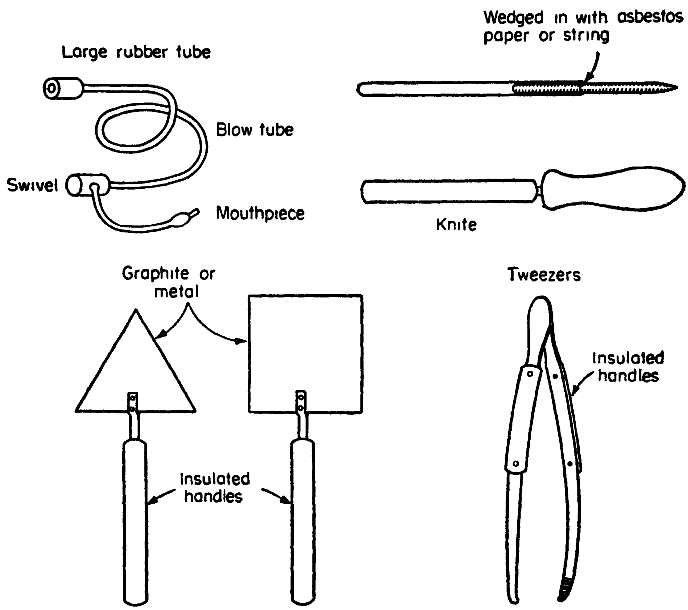


FIG. 8·1 Glass-Blowing Tools.

operation. Some torches are equipped with a variety of tip sizes. They will be found useful if the size of the work varies extensively.

A few other simple items (Fig. 8·1) should be at hand when working with glass. Many of these items may be obtained from scientific supply houses. They may be made up quite easily, however.

A "blow tube" in its simplest form consists of a few feet of rubber tubing about $\frac{5}{16}$ in. O.D. One end may be fitted with a mouthpiece and the other may have an adapter to make easier its connection to the tube to be worked. A "swivel joint" is

often used in the blow line. This enables the work to be rotated without twisting up the blow tube. Either a well-machined joint suitably greased or an ordinary packing joint gives a satisfactory seal, while allowing rotation. A supply of stoppers and asbestos paper tape should be in a convenient location. Some of the stoppers should be provided with a single hole and a tube which the blow hose will fit easily.

A carbon pencil is made by sharpening to pencil point shape a ¼-in. diameter carbon rod. This should be provided with a handle of glass or other insulating material. A sturdy pair of iron tweezers is very useful and may be provided with heat-insulating wooden handles or handles wrapped with asbestos string.

Brass paddles and pointed spades, usually made of about 0.030-in. brass and of various shapes, are often very useful. They should be warmed and rubbed in beeswax before being applied to the glass being worked.

Glass knives may be purchased or one may be made up by suitably grinding an old file. A piece of old broken grinding wheel makes a good stone for renewing the glass cutting properties of the knife.

It is essential to work on a bench or table top which will withstand the heat. Although asbestos board will serve the purpose, it does not wear well, and the asbestos-cement material known as Transite * makes a superior bench top.

8·2 Preparation of Glass

Before work with glass is actually started, a few fundamentals should be emphasized. Leave plenty of glass for "handles." Do not work the glass just at the softening point; better work can be done more easily when the glass is really hot. Always heat up a piece of glass slowly and cool it slowly. Do not keep the torch at one spot on the seal too long; keep the whole seal warm to avoid disastrous strains. Be sure all materials and tools are ready before starting. Think out the whole procedure before starting.

* Johns-Manville, 20 East 40th St., New York, N. Y.

The first glass blowing operation to be mastered is preparing the end of a piece of tubing to make a suitable seal. One method consists of heating the end of the tube to the fusion temperature, squeezing the walls together with tweezers, and pulling the resulting mass of glass to a long point. The torch is then used to cut this thread off, and the end is again heated as uniformly as possible. By using very gentle blowing pressure, this closed end is first just rounded over. Again uniform heating of the end is accomplished, and this time stronger pressure is applied so that the heated end suddenly blows out to a large bubble of paper-thin glass. This glass bubble is struck away, and the ragged

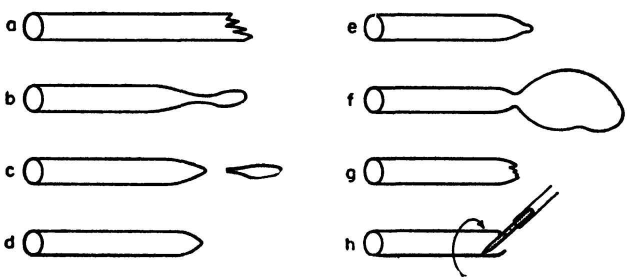


FIG. 8·2 Preparing an End.

edge left is smoothed down by fire polishing with the torch and rolling with the carbon pencil. Figure 8·2 shows the steps in the procedure. After considerable practice, it will be found possible to obtain good smooth ends by this method. Flared ends may be made by further rolling the end wall of the tubing out with a carbon pencil or a metal spade.

It should be mentioned again that, regardless of the type of glass used, it is good practice to introduce it to a flame gradually as in a series of passes through it. Likewise when the work is finished it should be allowed to cool in a similar fashion.

Actual breaking of the glass is sometimes a more convenient way of preparing tubing ends. It may be accomplished by a number of methods, depending upon the conditions encountered. The most simple and best known method, usable with smaller glass diameters, requires scratching the glass with the glass knife

at the place where the break is desired. Holding the glass firmly in the two hands, with the scratched portion at the far side and the thumbs opposite the scratch on the tubing, apply such force that the tendency is to pull the tubing apart and also to bring the ends toward you, putting tension in the scratch mark (Fig. 8·3*a*). This method nearly always works for small tubing, provided it does not have too heavy a wall. In case the tubing to

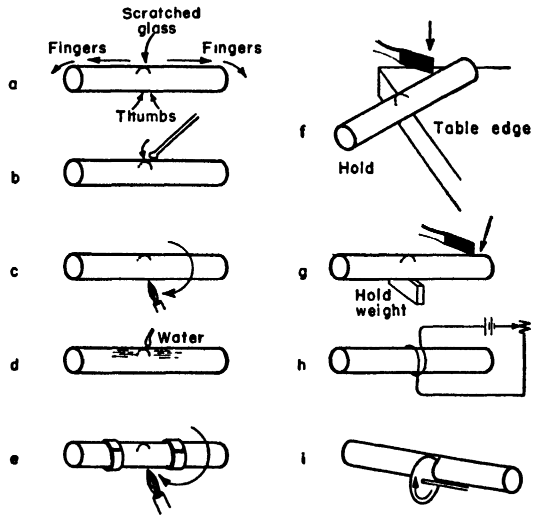


Fig. 8·3 Means of Severing Glass Tubing.

be broken is part of a rigid system, or for larger sizes of tubing, some of the following methods may be applied. A scratch mark is applied as before, and a piece of soft glass cane is heated in the torch until its end is a fused mass about to drop. This soft mass of hot glass is plunged against the scratch mark (Fig. 8·3*b*), and the resulting strains should give the desired fracture.

Another method consists of rotating the scratch mark of the tube over a small, sharp, pointed flame until the localized strains cause the glass to fracture (Fig. 8·3*c*). If the heat alone is not sufficient, a drop of water applied at the scratch after the heating process has progressed will often fracture the glass (Fig. 8·3*d*). The heating must not be carried too far, of course, or

the glass will merely soften. The idea is to create a highly strained ring of glass so quickly that it has no chance to relieve itself except by breaking.

In another method, starting again with the scratch mark, an evenly applied bandage of wet paper towel several layers thick and about half an inch wide is applied to each side of the scratch mark with a total separation of approximately half an inch (Fig. 8·3*e*). Rotation over the flame is nearly always successful with this method.

Figures 8·3*f* and 8·3*g* show methods of breaking tubing by tapping one end smartly while holding the tubing with its scratch mark opposite a table edge or the edge of heavy backing object. In the method of Fig. 8·3*h* current is passed through a wire of nichrome or similar heater wire suitably looped around the glass tube. The localized strains usually give a clean break by this method. Figure 8·3*i* illustrates the use of a thin abrasive wheel for cutting tubing.

When it is essential that the tube break with a good flat end structure the safest method is to carry the scratch mark completely around the glass where the break is desired. If an irregular end must be evened, the glass is heated to softening with the torch, after which the excess portions can be drawn away either with the tweezers or by use of a piece of cane glass which is stuck on the heated wall and withdrawn, carrying some of the unwanted glass with it.

8·3 Butt Joints or End Seals

The art of making butt joints probably can be best learned by starting with a size of glass roughly 3/8 in. O.D. Two techniques are possible here. One is to fix the torch rigidly, holding the two pieces of glass in the hands; the other is to work with a fixed or clamped piece of glass, holding the torch and the other tube in the hands. In either case one piece should be stoppered and the other made ready for blowing.

In case the torch is fixed, the two ends to be joined are brought into the flame at the same time. Here they are kept rolling simultaneously at the same speed. At first they are held a fraction of an inch apart, but as the temperature of the ends increases, they are brought closer together and fused just as the

glass softens. Before the temperature is reached, the flame will have taken on the bright yellow of sodium.

The rolling motion is continued, and at the same time the glass is pressed closer together. It will shrink inward, and should be allowed to thicken until it is roughly twice the normal wall thickness and the fusing has been so complete that the original line of demarcation has apparently vanished. The glass is then taken quickly from the flame and held in a vertical position to prevent sagging and uneven cooling. It is first drawn slightly to reduce the wall thickness and then blown to a little greater than the desired diameter and simultaneously drawn so that the outer diameter is uniform.

If this process is accomplished correctly, the result is a smooth seal having close to nominal wall thickness. It should be stressed

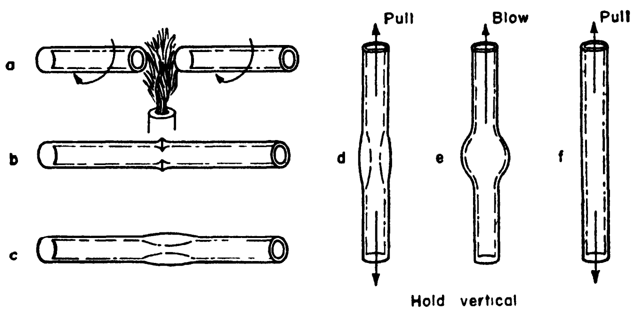


FIG. 8·4 Butt or End Seals.

that most consistent results are obtained if this sequence is followed: shrinking to thicken the walls, drawing and blowing, and then drawing again to give the proper diameter. Figure 8·4 shows the steps in making this seal.

In case the torch were held in the hand for this seal, exactly the same procedure is followed except that the simultaneous rotation of the pieces is avoided. The torch, however, must be moved around the glass so as to give uniform heating. Whenever possible in this case, the work should be arranged vertically since it is quite difficult to get a good horizontal butt seal without a heavier bottom wall because of sagging and running of the glass.

When glass sizes of much more than three-fourths of an inch are to be sealed, it may be found that the method of holding the torch in the hand will give better results. This does not necessarily hold true if regular glass blowing equipment, such as crossfires and blast fires, is available. Always in making a seal it will be found that extra time spent in properly preparing and shaping the surfaces which are to be sealed will be more than compensated for by the ease of making the seal. Uneven ends, for instance, usually give rise either to pinholes due to improper closure or to large masses of glass which are unsightly and which may also cause uneven annealing and possible fracture later.



Fig. 8·5 Sealing Large to Small Tubing.

These masses of glass may be reduced by "pulling" with a small glass rod. The torch is held mostly on the large mass of glass, keeping the entire work warm. Just as it softens, the glass rod is jabbed into the mass and a long thread pulled out. This thread is broken or melted off, and the process is repeated until the desired quantity of glass has been removed.

When two pieces of glass of different diameters are to be sealed the ends should first be worked to a common size. The smaller diameter tubing is heated until the end softens. By holding the carbon pencil just inside the end and pulling it against the wall as the tubing is rotated, a uniform flare is achieved. Where the diameters are not too widely different this matching is completely done by flaring the small tubing. However, it may be necessary to reduce the size of the larger tube also (Fig. 8·5). A certain amount of this may be achieved as a result of the natural drawing in of the glass when heated to the flow point. If this is insufficient, however, it may be desirable to heat a longer portion of this tubing and draw it to a smaller diameter, after which it may be cut with the torch at the proper point by the drawing and bubble blowing process described earlier.

8·4 Side Seals

The second seal to be mastered is the side seal or T seal. Figure 8·6 shows the steps in making this seal. In this case the tubing which is to be sealed to the side of the main tube has its

end prepared so that it is uniform and regular. Then at the point on the main tube where the seal is to be made, this entire region having been gently flamed first, a pin point flame is held on the glass until an area about equal to the radius of the tube has become yellow. Slight air pressure is applied at this time so that the glass just starts to bulge. The flame is then again applied over this area and the slight air pressure is again applied so that a bulge assumes a diameter about that of the tube to be attached. The center of this bulge is then heated and blown out to a large bubble. When the thin wall of the bubble

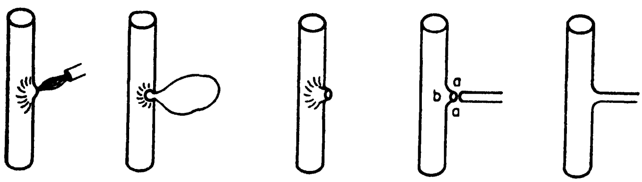


FIG. 8·6 The Side Seal.

is broken away there remains a properly positioned hole with a certain amount of side wall slightly smaller than the side tube in diameter. This hole is smoothed and opened to the right size with the carbon pencil.

Still using a fairly sharp flame, heat the two surfaces to be joined to the fusing temperature and stick them together. After the joining, the flame is first played on the portions of the joint in the recess formed by the two joining tubes (*aa* of Fig. 8·6). Each of these in turn is heated to a flowing temperature and blown out so that a good radius is achieved at the joint. The two sides (*b* of Fig. 8·6) are then sealed properly by fusing and blowing. The resulting joint should have a well-opened I.D. and present a minimum of restriction at the corners.

Often a hole must be made in a tube wall under circumstances which do not permit blowing the bulge and bubble. If the contained volume of the main tube is small it may be closed, and the pressure caused by gas expansion resulting when the desired hole position is heated may be sufficient to open the tube at the heated point. At other times the cane glass or a tungsten rod is again used. The wall is heated at the desired area, and a heated

cane glass is stuck against it and pulled away. In some instances the wall material may be pulled out to a cone which may be broken off, leaving an opening. In other cases the wall is merely made thinner and thinner by the repeated drawings until finally it opens up. In either case the carbon pencil may be inserted in the opening and, if desired, the hole reamed in such a way that a slight shoulder is thrown outward.

8·5 Ring Seals

The next type of seal is the ring seal. It is used many times in building simple devices such as water jackets, condensers, or certain types of traps. In this case, a glass of small diameter has its end flared and sealed to the inside of a larger tubing. The first step is to flare the inner glass properly. This is accomplished by heating the end uniformly with the torch. When the glass becomes sufficiently soft the carbon pencil is placed in the end at a slight angle. With the pencil held against the wall, the tubing is rolled quickly, giving a flaring action. For larger flares, it is usually necessary to repeat this process several times. A little experience is necessary to get the enlarged diameter so that the flare will just easily slide in the larger tube. If the flare is accidentally made too large it may be reduced by rolling it on a brass paddle. Often the flare will also be eccentric with respect to its tube and possibly far from circular. A certain amount of this may be tolerated as it may be taken care of in making the seal.

Assuming that a suitable flare has been obtained, the small tube is inserted in the larger until the flare comes to the place where the seal is to be made. The free end of the small tubing is then held centered by means of a rod in a one-hole stopper or by means of some material, such as asbestos, wrapped around the tubing. All the open ends are then closed with the exception of one for the blow tube. The supports should be arranged so that after the glass is joined blowing pressure will reach both sides of the seal inside the tube. This combination is then rotated over the torch so that the flame plays on the region surrounding the flared edge. Figure 8·7 shows the steps in making this seal.

As the glass heats up, it will shrink to a smaller diameter and make contact with the flare. The heating is continued, as is the

continuous rolling of the tubing, and as the glass softens and fuses to a good seal the two ends are compressed slightly so as to thicken the glass in the seal and bring the diameter at this point to the normal value. Some improvement may be achieved in appearance and uniformity by counteracting the glass shrinkage with gentle pressure in the blowing tube at proper intervals.

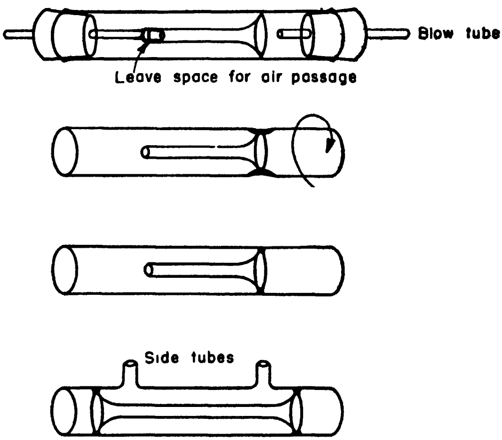


FIG. 8·7 Ring Seals or In-seals.

The first of such seals is usually not difficult to place in a piece of apparatus, but quite often it is desirable to form a second with the other end of the inner tube so as to provide a hollow wall, for a water jacket, for instance. In this case, the two flared ends are prepared at the same time. After the first ring seal is made, an opening in the side wall must be provided before the second seal is made, to avoid building up a pressure in the hollow wall. Since a side tube is quite often desired near each end of such equipment, as for water circulation of a condenser, this side tube should be sealed on while the glass is hot from its adjacent ring seal. This side tube will then serve to prevent pressure formation.

It is usually quite difficult to complete two ring seals close together. The different amount of expansion on the inner and outer tubes due to temperature differences gives rise to strong forces which may cause fracture after cooling. To minimize this

effect, as great as possible a separation of the seals should be attempted and heating of more than the amount of glass necessary to make the seal should be avoided.

When it is desired that the small tubing diameters be carried past the ring seal the procedure shown in Fig. 8·8 is convenient. The ring seal is first made against the closed end tube. This closed end is then blown out and a butt seal made to it with the smaller tubing.

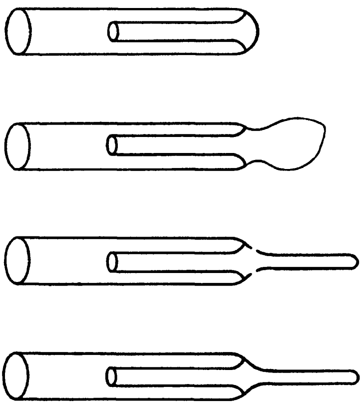


FIG. 8·8 End Seal to Ring Seal.

In doing any glass work, proper annealing procedures should be observed. In Pyrex when a seal is finished it is usually necessary to spend only a minute or so with a strong brush flame to bring the temperature of the glass to a safe value. Soft glasses, however, like the lead or soda glasses, require much more care. They must be cooled down, with the flame constantly being softened, until finally the ordinary gas flame is arrived at and carbon is allowed to deposit over the joint. The best annealing, of course, is obtained with a well-controlled oven. However, this is seldom possible with the joints made in glass systems.

It is very difficult to obtain good bends in tubing by using a small torch because to make such bends requires the heating of a rather long portion of the tubing. Fairly good bends may be made, however, by heating the longest portion possible and keeping a large radius. Wrinkles and flat portions must then be worked out by heating and blowing. It is many times difficult to make a bend in which all the glass lies in a common plane. One method is to allow one end of the tube to fall under gravity while the glass is being heated at those regions indicated by the bending of the glass and the desired shape. When a short radius bend is required it may sometimes be most satisfactorily achieved by making a side seal and then removing the excess arm of glass.

Although many metal-to-glass seals, such as small cylinders and eyelets and the Housekeeper type (feather-edge copper seals), are of a size which permit working with a hand torch, these seals will not be described here as they may be obtained commercially. The so-called graded seals, in which a number of intermediate seals are used to join materials having widely separated coefficients of expansion, may also be obtained commercially.

8·6 Constriction for Seal-off

At times it is necessary to separate physically two parts of a glass system while maintaining the vacuum in each. Examples are radio tubes, lamp bulbs, and thermos bottles, which must

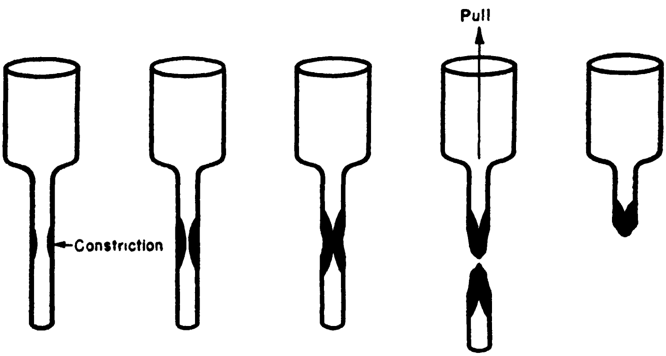


FIG. 8·9 Constriction and Seal-off.

be sealed-off for removal from the vacuum system. This sealing-off process should be anticipated in the assembly of the device and an exhaust tubulation provided in the glass envelope. In some cases it may be possible to locate the tubulation so that the remaining stub of glass will be out of sight and also protected. The seal-off tips of radio tubes and lamp bulbs are covered by the cemented-on bases.

The sealing-off process (Fig. 8·9) consists of uniformly heating a very short length of the exhaust tubulation, causing it to shrink inwardly until it finally closes completely, thus sealing one part from the other. At the same time, if one part is care-

fully pulled from the other, the glass necks down and finally parts. The torch is then played on the resulting pointed end of glass, rounding it over and making breakage less likely. Very careful flame annealing is required because of the thick glass. A very small flame is required to keep the amount of heated tubing to a minimum and thus prevent the liberation of excess gas.

When evacuated thin-walled tubing is heated there is a tendency for the glass to suck in and rupture. With tubing under one-fourth inch in diameter the ratio of wall thickness to inside diameter is such that this danger is not too great and good seals can be made easily. For larger glass tubing it is customary to thicken the exhaust tubulation to a constriction at the point where it is planned to make the seal-off. This is shown in Fig. 8·9.

The constriction may be made either before or after the tubulation is sealed to the tube. In any case it is essential that one end be free so that the tube can shorten as it thickens. The operation is simple if the tubing can be rotated in a flame. The vertical position is best if the tubing cannot be rotated as the natural sagging of the heated glass is thus used to help build up a uniform wall.

Although pumping speed is usually not so important in sealed-off tubes, the placing of such a constriction will markedly affect the pumping time and it is wise to leave the constriction as large in diameter as is consistent with safe seal-off. The best constriction in a given case will depend upon the type of glass, the diameter of the tubing, and the operator's experience. A good general rule is to hold the wall thickness of the constriction about equal to the constriction diameter.

8·7 Small Glass-Metal Seals

Although the larger metal-glass seals will not be discussed here, the use of sealed-in wires is so common and the technique so useful that a few methods will be described. Table I shown earlier in the chapter gives the metal-glass combinations which have proved most dependable.

It has been found most satisfactory for most sealed-in wires to "bead" the wire first. The basic procedure followed is to

clean the wire and oxidize it where necessary, immediately after which a short length of tube of "beading" or "wetting" glass is slipped over the wire and fused on. This beading process is carried on by rotating the work in a small pointed flame. A suitable pin vise is convenient for holding the wire for this operation. The glass is shrunk down onto the wire, care being taken to work from one end to the other so that no gas is trapped in the bead (Fig. 8·10). The quality of the metal-glass seal can easily be determined by inspection of the bead. A good seal to Dumet should show cherry red, tungsten should be straw colored, and Fernico or Kovar should be gray.

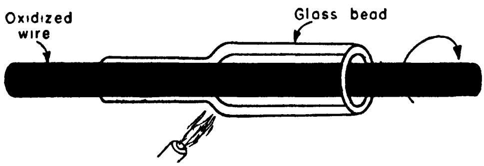


FIG. 8·10 Beading a Wire.

The beaded wire may be sealed through a side tube as shown in Fig. 8·11 or if a number of leads are to be used, as in the case of a radio tube stem, they are usually assembled in a press as shown in Fig. 8·12. In this case the wires after beading are arranged in their proper positions and a cross-wire which may be removed later is spot-welded to them to hold them together. (When a large number of identical stems are to be made it may be desirable to make up a more convenient holder for the beaded wires.) A partially flattened "flare" is then slid over the assembly and the glass heated. When it is softened the glass is squeezed down firmly over the beads with a pair of pliers or heavy tongs forming a "press." Care must be taken to get a good seal to all the beads or a hairline leak will result. The stem must be annealed carefully, after which it may be fitted with its electrodes and sealed to the vacuum system. In the manufacture of radio tubes and lamps this same general procedure is followed, the operations being performed automatically.

If the leads are very small the seal may be made through the glass wall of a piece of tubing so that it is flush with the

wall on both sides. Such seals are best made with ribbons of platinum foil, say about 0.020 in. wide and 0.0005 in. thick, or with tungsten wire less than about 0.008 in. or 0.010 in. in

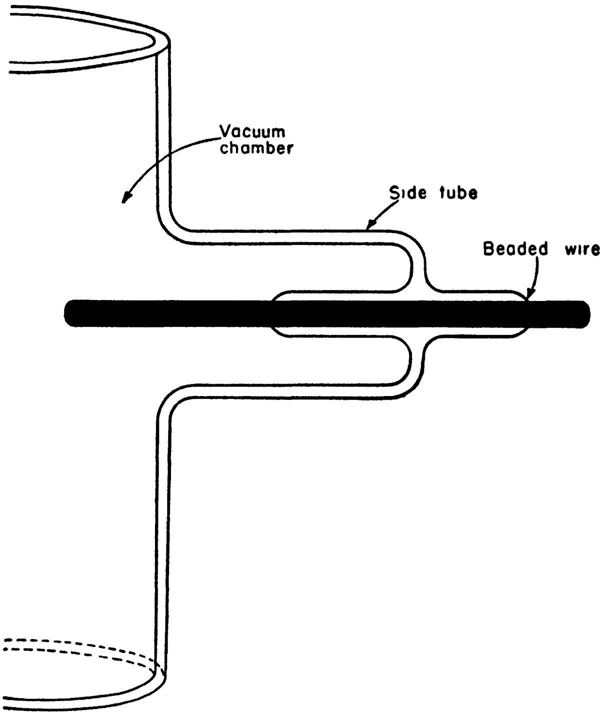


Fig. 8·11 Beaded Wire Sealed in Tube.

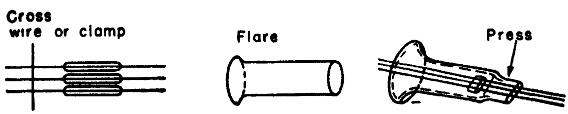


Fig. 8·12 Steps in Making a Flared Press Seal or "Stem."

diameter. If tungsten is used it should be beaded extremely thin.

In making this seal a small hole just large enough to admit the ribbon or wire is drawn in the glass wall (Fig. 8·13). The

ribbon is then placed through the hole and pressed firmly against one side while the region is heated with a small pointed flame. Care must be taken that the ribbon is not melted away in getting the glass molten for the seal. After the ribbon is stuck well to one side the remainder of the hole is pushed down and sealed, giving an outside flush surface as in *d* of Fig. 8·13. The glass is now heated from the outside and the ribbon extending inward is pushed up against the inner wall as in *e* of Fig. 8·13.

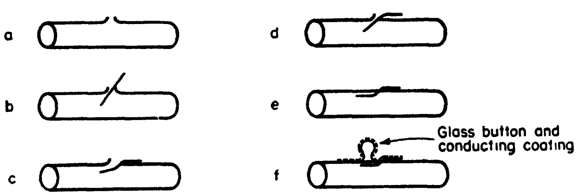


FIG. 8·13 Sealing Small Ribbon or Wire Through Glass Wall.

Connections may be made to such seals with silver paste or other suitable coatings which are described in the next chapter. To provide a better means of connecting to such a seal, a small piece of cane glass may be fused to the wall near the externally exposed ribbon as shown in *f* of Fig. 8·13. When they are both coated with conducting material connections may be made with a battery clip or by wrapping bare wire around the projecting glass.

Dumet wire was developed to take the place of platinum for the soft glass applications as in lamp bulbs and radio tubes. This wire has a nickel-iron core in a copper sleeve. It may be obtained commercially with various other metals butt-welded to its ends to facilitate its use. Its use is not recommended in sizes much larger than about 0.020 inch because of its axial and radial expansion characteristics.

Tungsten may be cleaned for beading by heating it to bright red and drawing it while hot through a stick of sodium or potassium hydroxide, after which it is washed in distilled water. It is then oxidized and immediately beaded. Better tungsten-hard glass seals are made by using either Nonex or 705 glass as a wetting glass. Such a beaded wire may then be sealed di-

rectly to Nonex, which in turn can be sealed to Pyrex. Although Nonex to Pyrex seals can be made it is not good practice, particularly in larger seals, and an intermediate glass is desirable. Corning glasses, Uranium canary glass 3320, and blue glass 5420 or 707 are suitable intermediate glasses.

Tungsten has a characteristic which should never be forgotten when using it as a conductor through glass. Tungsten wire many times splits in a direction parallel to its axis. Such a leak may be extremely difficult to find since it is very small and quite often it apparently opens and closes with the passage of current through the wire. This defect is most often encountered with larger sizes of wire.

The trouble has been eliminated by filling the recesses with copper,[2] but in general it is sufficient to specify "for sealing to glass" when ordering tungsten and to take care that the wire not be bent or twisted prior to its use.

Fernico or Kovar should always be normalized before it is used in a glass-metal seal. Normalization is accomplished by bringing the metal up to 1100°C in a hydrogen atmosphere. After 15 minutes at this temperature it may be cooled at any desired rate and removed from the hydrogen atmosphere, after which it may be used.

8·8 Rhodium-Coated Electrodes

Many times glass work must be done close to a metal electrode. The result is usually a badly oxidized electrode. A method of minimizing this oxidation to a great extent is to provide the electrode with a coating of rhodium. It is possible to shrink a Pyrex tube down over a rhodium-coated molybdenum disk without causing discoloration due to oxidation. The rhodium may be applied by evaporation.

8·9 Hole Patching in Glass

Pinholes in glass may be sealed up using a drawn thread of glass or a pointed glass rod. The thread and the work are both heated and, as the thread softens, it is plugged onto the pinhole and the excess pulled away. The entire region is then heated until the added glass fuses well over the hole and into the surrounding region.

In closing larger holes the glass thread may be laid back and forth across the hole until it is closed, after which it is fused to form a smooth wall. In another method, a small tube is flared until it covers the hole. After sealing in a manner similar to that used in making side seals the tubing is shrunk down and

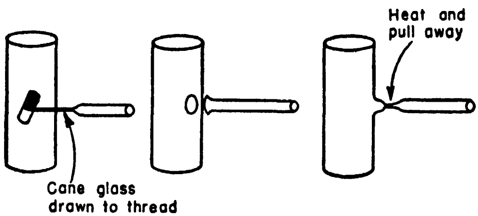


FIG. 8·14 Methods of Patching Holes.

pulled away and the region worked to a smooth wall. These methods are shown in Fig. 8·14.

At times there is sufficient material around a pinhole or crack, but heating tends to enlarge the opening. It may be possible to push the softened edges together with a carbon pencil or draw them together with unheated cane. A pointed tungsten rod is convenient for such occasions since it will stick in the molten glass well enough to allow pulling it together, but easily breaks free of the glass as it cools.

REFERENCES

1. W. J. SCOTT, *J. Sci. Instruments,* Vol. 23, pp. 193–202, September 1946.
2. A. L. REIMANN, *J. Sci. Instruments,* Vol. 23, pp. 121–124, June 1946.

Chapter 9

VACUUM SYSTEM TECHNIQUES

9·1 General Discussion

Up to this point the many devices and components from which a system may be assembled for removing the air from testing chambers or for controlling or measuring the gas pressures in such a system have been discussed. Now the matter of the assembly of such a system for application to experimental electronics, together with some techniques that are quite useful, will be taken up.

Vacuum systems may be divided first of all into either glass or metal systems. In addition, a further division may be made between those to be used temporarily for certain tests, which may be called demountable, and those arrangements from which are to be obtained sealed-off tubes.

It might be thought that sealed-off tubes would have to be classified as production rather than experimental. Actually, however, in certain instances it is possible to perform the experiment only if a sealed-off tube and the techniques associated with such a tube are used. For example, it may sometimes be expeditious to obtain a desired result by making several complete structures varying only slightly from each other rather than to build this variability into demountable experimental equipment.

Again, in connection with photo cell experiments, or indeed most work dealing with sensitized surfaces, a number of sealed-off structures may yield comparative results more consistently than more complicated demountable apparatus, which, although it provides easy access to the components, also may prove a source of contamination. Actually, one usually does not draw the line between any of these definitions but uses the arrangement most suitable to the experiment at hand.

Probably the most convenient type of system consists of one fundamentally of glass, preferably Pyrex. In the techniques to be discussed, a Pyrex system shall be considered the backbone of the combination, and where components of other materials are introduced they will be specifically designated. It is probably quite true that almost anything done in a glass system can be done equally well in a metal system. Certain adjustments must be made in the techniques in the two cases, however, and although this discussion will be based upon a Pyrex system, its use will later be compared with metal as regards the advantages and disadvantages and the available components and techniques in the two cases.

9·2 Supports for Components

Considerable care must be exercised in the assembly of a glass system to insure that support is provided at the proper places but that, on the other hand, the structure is not so rigidly held that a slight vibration will set up strains resulting in fracture. Wherever glass is to be clamped it should first be surrounded with material such as cork, asbestos tape, rubber tape, or rubber tubing. The use of asbestos tape makes possible flaming of the glass if this is necessary. The clamp may then be pulled down to give a support which, although it is quite firm, still will allow some movement of the glass.

When a piece of apparatus such as a pump requires support at two or more places the clamps should be tightened a little at a time and occasionally loosened as a check. A dangerous condition of strain is indicated in such a mounting if there is a noticeable shift of the glass when one clamp is loosened. Provided the heavy components are supported so that no swinging action may occur, a general rule might be that fewer clamps on a system are better than many. Extensive sections of glass tubing allow considerable flexing and thus compensate for any small vibrations if allowed to do so. If rigidly confined, however, this compensation cannot take place and the slight jar may result in breakage. Figure 9·1 shows a number of means of supporting glass apparatus.

Although moistening a rubber tube will aid in slipping it over a piece of glass or metal tubing, the rubber will subsequently

"freeze" and be difficult to remove. Castor oil is a very good lubricant for rubber in this instance. An excess should be avoided as it slowly creeps along the tubing and may be drawn into parts of the system as a contaminant. Many hands have been badly cut by glass tubing that broke under the tight grip

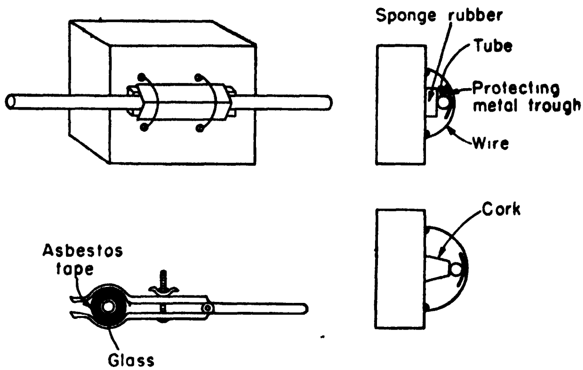


FIG. 9·1 Means of Supporting Glass Tubing.

and twisting motion necessary to force rubber tubing on glass or to remove it. In such cases the hand should be protected by a handful of cloth or a stout glove.

9·3 Attenuation of Vacuum, Placing of Valves and Gauges

A certain amount of time is required for gas to migrate from the far reaches of a system to the vacuum port. As a result many regions will be found which have pressures appreciably higher than that obtained near the pump. Everything else being equal, the more remote the region the poorer the vacuum.

The limitations imposed upon a pumping system by the use of great lengths of connecting tubing or tubing of small diameter have been recognized since the early days of vacuum work. However, many vacuum systems apparently are assembled with little regard for these limitations. It is quite commonly assumed that bends and variations in diameter reduce the conductance of a tube. This is not true unless an actual restriction is introduced.[1]

It is probably quite true that in many vacuum systems the design is dictated by the equipment that is available. An assembly of woefully mismatched pumps and connecting tubing which is furnishing a vacuum suitable for the experiment may be justified in a temporary laboratory set-up, but where there is a choice of components an attempt should be made to match the components to the job. This is not always easy to do since the requirements as well as the conditions are many times variable.

In some cases it is desirable to evacuate a large volume to some medium vacuum as rapidly as possible. On the other hand it may be necessary to evacuate the same volume to the best possible vacuum regardless of time. Again this same volume may be provided with a contaminating influence which will continuously evolve large quantities of gas. Each of these different sets of conditions requires its own treatment.

A few fundamental concepts should be remembered, and their application, together with the use of good judgment based on experience, will make possible the assembly of good practical systems.

In a system containing side tubes and restricting shapes and in which there may be located some contamination resulting in a virtual leak we may find that the vacuum gradient is not simply a function of the distance through which the gas must be pumped. There are instances where a special exhaust port should be provided near a high-pressure region even though the rest of the system is being pumped by the standard vacuum equipment.

In many cases identical gauges attached to various parts of the system will vary in their readings by as much as ten to one. In the design of a piece of experimental equipment this fact must be kept in mind. Exhaust ports should be placed so that the best vacuum is obtained where it is most essential, and the values read from the gauges must be interpreted as dictated by experience rather than taken to represent accurately the entire system. Similarly, the placement of valves or controlled leaks should take these factors into consideration. When gas is to be admitted to a system in some controlled manner, it should be remembered that although it will gradually diffuse outward through the whole system, in general there will be a main flow

toward the pumps with some variations introduced due to the configuration of the system and its contents.

Diffusion pumps are usually much faster than their accompanying rotary pumps in the region where they first take hold. However, they contribute nothing until the pressure is reduced to some value usually well below 100 microns. When pumping time is of interest there are two phases of the pumping operation to consider: that in which the rotary pump is removing gas at relatively high pressures and that in which the vapor pump is removing rarefied gas and the rotary pump in acting as a backing pump is usually not heavily loaded.

The physical concept responsible in part for this situation is that of the mean free path of the gas, that is, the average distance which a gas particle moves between collisions. If the pressure is such that the gas molecules in their random motions collide many times with each other before striking a container wall the mean free path is said to be smaller than the geometry of the container. On the other hand, the gas may be rarefied to the extent that between collisions the molecules travel great distances and thus may strike the walls as frequently or more frequently than they strike other molecules. In this case the mean free path is greater than the geometry of the container.

For a molecule moving among gas molecules of its own kind, the mean free path is inversely proportional to the square of the mean molecular diameter and to the number of molecules per unit volume, that is, the gas pressure. The gases commonly encountered in experimental electronic work have mean molecular diameters ranging from about 2×10^{-8} cm to 4×10^{-8} cm. The mean free paths of nitrogen (approximate diameter of 3.5×10^{-8} cm) for a number of pressures is shown in Table I.

TABLE I

MEAN FREE PATHS OF NITROGEN AT VARIOUS PRESSURES

Gas Pressure (mm Hg)	*MFP (cm)*
760	6.7×10^{-6}
1	5.1×10^{-3}
10^{-3}	5.1
10^{-5}	500

Because of experimental uncertainties and theoretical assumptions no particular emphasis should be placed upon the exact numerical values in the table. The orders of magnitude should be kept in mind, however. In particular it should be noted that at gas pressures around 10^{-3} mm Hg the mean free path is of the order of magnitude of the geometry of the tubing diameter and spacings of most experimental vacuum arrangements. It should be mentioned here that from kinetic theory concepts the mean free path of an electron moving through a gas is greater than that of a molecule of the gas by a factor of $4\sqrt{2}$.

When the mean free path of a gas is small compared to the diameter of the tubing through which it flows the laws governing its flow are hydrodynamic in character and the flow is proportional to the pressure. As has been pointed out this condition occurs in ordinary laboratory vacuum systems at pressures above a few microns.

A pressure gradient exists along a tubing connecting a region of high pressure to one of low pressure. Such a situation occurs, for example, when a forepump commences to exhaust a large vacuum system. Under such conditions the rate of flow of gas through a cylindrical tube of length L and diameter D is governed by the relation

$$\text{Volume of gas per unit time} \propto \frac{D^4}{L}(P_2^2 - P_1^2) \qquad (9 \cdot 1)$$

where P_2 and P_1 are the high and low pressures respectively.

When the mean free path is greater than the tubing diameter the gas flow is molecular in nature and no longer is a function of the pressure. Under such conditions reference may be made to the "speed" of a piece of tubing just as to the speed of a pump. In fact the speed of a system can be calculated approximately by considering the various speeds of the components as conductances in electrical theory and combining them according to electrical laws.

For straight vacuum tubing at these low pressures the speed is governed by the relation

$$S \propto \frac{D^3}{L} \qquad (9 \cdot 2)$$

where S is the speed of the tubing and D and L are the diameter and length respectively.

We have already seen in section 2·1 that the speed of an aperture, such as a hole in the vacuum wall, is a direct function of its area, giving the relation

$$S \propto D^2 \tag{9·3}$$

where S is the aperture "speed" and D is the aperture diameter.

Detailed formulae exist[2] for the precise determination of pumping speed characteristics of tubing and combinations of tubing and pumps. They are not included here for two reasons. First, except in the most simple cases the final calculations must be adjusted by a factor, usually of several fold, which can be determined only by experience or actual test; second, in the average laboratory it is usually more desirable to use available auxiliary equipment if it will serve, even though it is mismatched.

Therefore, although calculated vacuum systems are not decried (in fact, they are readily endorsed), it is accepted that on most experimental vacuum systems the calculations will be omitted. However, in assembling a system one should bear in mind the influence of the tubing diameter on pumping speed and gauge readings as shown in the foregoing relations. There are times, for instance, when the improvement factor of 8 gained by using one-inch tubing instead of half-inch tubing is of importance. On the other hand, if the volume to be exhausted is very small compared to the volume of the tubing, increasing the tubing size may be ridiculous.

It will be apparent that there will be very few occasions when a chosen set of pumps will be ideal for all phases of the exhaust of a system. For instance, a system having large volume, but which is tight, will require a large-capacity rotary pump to exhaust it in a reasonable time to where the vapor pump takes over. After this point is reached a much smaller forepump would suffice. Such arrangements are quite frequent. Either larger pumps or extra pumps are used at first, and then by means of valves they are removed, leaving a unit of suitable capacity to carry the load.

In some instances with smaller vacuum systems which are tight and may be baked out it is possible to do away with the

forepump completely for long intervals. In such cases a chamber, usually of glass, having a comparatively large volume is evacuated and then arranged to take the exhaust of the vapor pump. This arrangement will function until the gas being pumped into the chamber builds up to a pressure which no longer allows the vapor pump to operate. Usually the chamber is merely placed in the vacuum line between the rotary and

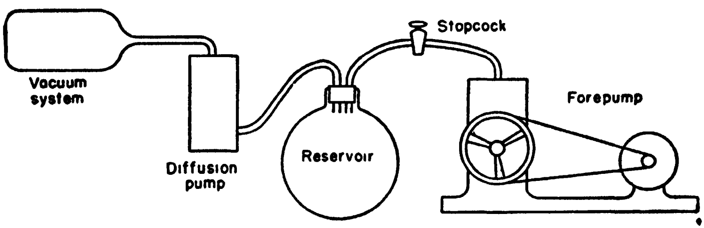


FIG. 9·2 Use of Reservoir in Forepump Line.

vapor pumps with a valve arranged between it and the rotary pump (Fig. 9·2). With suitably balanced components it is sufficient to run the rotary pump a short while once or twice a day.

Some vacuum systems require frequent opening to air and it may be undesirable that so much air pass over the hot pump fluid. By-pass valves are often used to eliminate this factor. They isolate the vapor pump until the system is evacuated to a satisfactory value by the rotary pump, at which time the vapor pump, ready for operation, is again placed in the line.

9·4 Cleaning of Parts

In the cleaning of parts to be used in a vacuum system a little experience and the exercise of good judgment may save much time and labor and indeed may often save the experiment from failure. In general, it is better to err in favor of extra cleanliness. However, this may be overdone. For example, it is neither necessary nor desirable to exercise in all cases the extreme care and meticulous cleanliness necessary in experimenting with conditioned surfaces as in studies of photoelectric and secondary emitting surfaces.

It should not be necessary to point out that in discussing cleanliness it is assumed that gross contamination, such as visible de-

posits, excess grease, or scale, is always removed. In electronic vacuum practice the cleaning problem consists first of eliminating sources of gas which might act as virtual leaks and so make difficult the achieving of a suitable vacuum, and second removing materials which might affect the experiment because of their physical or chemical nature either *in situ* or as a result of vaporization or migration.

For ordinary electronic vacuum practice it is usually sufficient that a metal part be degreased and hydrogen-fired before use. The degreasing procedure may consist of washing and rinsing in a good degreasing agent such as carbon tetrachloride, ethylene dichloride, or ether. It is not sufficient just to dip a part in the solvent. Grease taken into the solvent from a small speck of solid contaminant at once becomes diffused through the solvent. If the part is merely dipped and then allowed to dry the result is a thin film of grease completely covering the part. Good practice requires, in addition to the dipping, several rinsings with clean solvent.

Commercial degreasing units heat the solvent, and much of the degreasing action is carried on in the hot vapors. Provision is made for self-cleaning of the solvent by a continuous process of distillation. Precautions should be taken with all organic solvents that the fumes not be inhaled as severe poisoning may result.

Hydrogen firing is a very effective means of removing oxides from most metals. Hydrogen of commercial quality is satisfactory for ordinary metals, but those alloys containing chromium, such as nichrome and stainless steel, will usually not clean properly unless the last trace of water vapor is removed from the hydrogen. This may be accomplished by a number of methods. Filters containing any of the well-known chemical drying agents are helpful. Liquid air or CO_2 traps and filters containing freshly reduced copper chips heated to about 350°C are also beneficial. Usually some combination of these methods proves satisfactory.

Hydrogen furnaces, which vary in size from a few inches to many feet in diameter, usually consist of long tunnel-like chambers. Such a chamber is continuously furnished hydrogen, which escapes at the ends and there is usually kept burning.

The central portion of the chamber is held at the desired temperature. Materials to be fired are fed in one end of the chamber, sometimes in shallow containers called "boats," and at the desired rate they are fed through the furnace. Sufficient chamber length at the exit end allows for slow cooling in the hydrogen atmosphere. Temperatures in the neighborhood of 1000°C are usually sufficient, but lower values are sometimes dictated by the nature of the material.

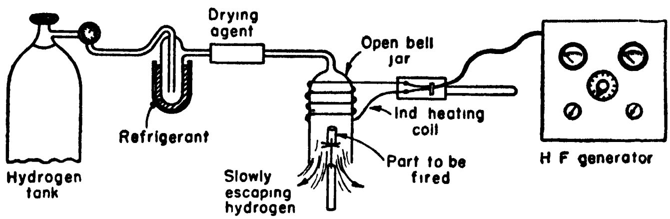


Fig. 9·3 Open Bell Jar Hydrogen Firing Apparatus.

A very satisfactory arrangement for firing small parts is shown in Fig. 9·3. Hydrogen is fed through the drying train into the top of the vertical hydrogen bottle, which has its bottom end open. The flow of hydrogen is adjusted so that it just may be positively felt by closing the bottom of the tube with the cupped hand. The outflowing hydrogen is not ignited in this case. The induction coil is arranged around the tube well up from the open end. It should not touch the glass tube, which preferably should be of hard glass, or localized heating may crack the glass and possibly cause the tube to explode. A shield of mica or a concentric portion of glass tubing serves to prevent this contact.

After the hydrogen flow is adjusted the parts to be fired are raised up in the tube into the region of the induction coil. After waiting a few seconds for the hydrogen flow to wash out any air introduced, the induction coil may be energized and the power adjusted to give the desired temperature to the part. Further control of the heating may be accomplished by adjustment of the part in relation to the coil.

Such parts as cylinders, disks, and other such shapes may be easily heated by induction from the coil and may be held in

position in the bottle by a suitable hook or wire frame attached to a long handle of glass or other suitable insulator. Wires, small parts or shapes not suitable for induction heating in themselves, may be held in a deep thick-walled metal cup fastened to a glass handle. The iron shell of a metal radio tube makes a very suitable container for this purpose. When firing small parts in this way care should be taken to insure removal of the introduced air. The bottom of the metal container should have a few small holes to facilitate this removal. The parts should be allowed to cool before removal from the hydrogen atmosphere.

For satisfactory results an induction unit of at least 3-kw capacity should be available. The arrangement described is essentially a temporary laboratory set-up and care is essential for its safe and satisfactory operation. Provision should be made to prevent the escaping hydrogen from accumulating to dangerous proportions. Suitable screens or shields should be provided to protect the operator in case of explosion of the bottle which might occur if the hydrogen flow decreased, allowing air to get in near the heated parts, or if the tube cracked for some reason.

If it is desired to fire the parts in vacuum rather than hydrogen, a similar arrangement may be used. The open mouth of the bottle is fitted with an easily removable cover using grease, wax, or rubber gaskets, and the opening normally used to admit hydrogen is connected to the pumps. Since the parts cannot now be held in position from the outside with the glass-handled holders other positioning means must be devised. This may be made easier by the fact that it is no longer necessary to keep the tube in the vertical position as was dictated by the condition of hydrogen flow in the case of hydrogen firing.

The process of hydrogen or vacuum firing is often useful in relieving strains in metal parts. The normalizing of Fernico or Kovar was mentioned in Section 8·7. Operations such as bending and spot welding, such as are used in building an assembly of electrodes, usually introduce strains in the structure which may be relieved by strain annealing by hydrogen or vacuum firing.

There are times when a part may not lend itself to cleaning by means described above. For instance, it may be required to give a final cleaning to an entire structure such as a radio tube

mount prior to its being sealed into a bulb. Such a structure may contain glass, mica, ceramic insulators, and cathode coating in addition to all the metal parts. The following technique has proved good. The unit is first dipped and rinsed in boiling dilute acetic acid. This process is then repeated with boiling distilled water and finally the part is thoroughly rinsed in pure alcohol. Drying may be accelerated by storage in a warm cabinet. A few lamps kept lighted in a cabinet usually provide sufficient warmth.

The commonly used insulators, mica, glass, porcelain, Mycalex, and talc,* may be given any of the degreasing or washing treatments described above. Likewise, they may be fired in an inert atmosphere, but like metals certain temperature limits must not be exceeded. Mica can be fired safely up to about 700°C and should always be baked before use to remove water vapor. Mycalex, a mixture of powdered mica and soft glass, is limited by the temperature which can be withstood by the glass—between 300° and 400°C.

Porcelain and prefired talc will safely withstand any normal firing temperature. Natural unfired talc, however, must be fired very carefully. Its expansion characteristics are likely to be unpredictable and commonly are different in different directions. This is important to remember because it is frequently necessary to fabricate an insulator from unfired talc, which machines very easily. The part is then fired, after which it can be worked only by grinding.

The harder insulators such as porcelain, glass, or fired talc may oftentimes be cleaned satisfactorily by sand blasting. Fresh clean sand should be used, and it should be remembered that such blasting removes an appreciable amount of material.

Natural rubber such as is used in vacuum hose or gaskets may be cleaned by washing thoroughly in boiling caustic soda solution. Tubing may be cleaned by holding the ends with tongs and alternately raising and lowering the ends, allowing the solution to flow through. This cleaning should be followed by a thorough rinsing in distilled water and finally the rubber should be well dried.

* The trade name Lava has been given by industry to talc or soapstone, which is the natural stone form of magnesium silicate. The term has also been extended to include the stone form of aluminum silicate.

Where only moderate cleaning procedures are being followed, as in the case of some continuously pumped apparatus, the parts may be handled with bare hands. Regardless of how well scrubbed the hands are, however, it is only a short time before they are again lubricated with natural oils and, although not ordinarily visible, such oil spreads out immediately over a well-cleaned part from the point of contact of the fingers. Perspiration from the hands may also cause much trouble if it is allowed to contaminate the parts of an electronic device. Salt introduced in this way can cause the device to fail because of low electronic emission from the cathode.

Therefore, when real cleanliness is essential lintless gloves are necessary. Rubber gloves are satisfactory as they may be cleaned easily. For long periods of wear, however, washable white cotton gloves are customarily used.

In the study of surface phenomena and sensitized layers the cleaning procedure is quite often the most complicated and exacting part of the experiment. The cleaning of surfaces for such work, although so highly specialized, is based upon the fundamentals described here.

9·5 Degassing

Any material will be found to contain a certain amount of gas either on its surface or within the material itself. That gas which is occluded on the surface may be removed by several methods; that contained in the material makes other treatments necessary. When a material is placed in a vacuum there is, of course, an immediate tendency for its gases to diffuse from it into the surrounding vacuum. The amount of this loss of gas is for most materials very small at room temperatures, although it varies widely from material to material and depends upon the nature of the gas.

The most common method of speeding up this outgassing is to raise the temperature of the material. The return is increased tremendously by this means, and it becomes more and more effective as the softening or melting point of the material is approached. Usually a baking-out process is not carried to this extreme as it is necessary to maintain the original physical characteristics of the material. Also, since many of the materials

will not be operated much above room temperature, it is necessary only to bake them at some temperature well above their operating temperature in order to prevent their acting as virtual leaks under normal operating conditions.

The outgassing by means of raised temperature, of course, must be done in the presence of vacuum for greatest efficiency. However, there are times when it is desirable to heat a material in an atmosphere of some gas, as say, hydrogen, in which case the hydrogen to a great extent replaces the other gases of the material. If the object is then introduced into the vacuum system, the hydrogen may be removed much more readily than the original gas. Also, as described in the preceding section, hydrogen firing and degreasing are standard procedures for eliminating contaminating gas sources before their admission to the vacuum system.

In the case of all-glass systems or all-metal systems it is common practice to provide with the exhaust equipment a bake-out oven which may be electric or gas. Some arrangement is made to maintain accurate control of the temperature. Such ovens are usually arranged overhead with counterweights and pulleys or are otherwise made easily removable so that they can be introduced in the processing at the strategic time and removed when their job is finished.

Such ovens may be made up easily. They are usually constructed either with well-insulated walls or of polished metal such as aluminum with no insulation at all. The insulated type is usually made up of an angle iron frame fitted with asbestos sheet walls ½ to 1 in. thick. The concrete-asbestos board known as Transite,* although fairly flame resistant, is of little use as a heat insulator. True asbestos board should be used. A more elegant type of furnace consists of two concentric metal shells, the intervening space being provided with suitable insulating material.

If gas is used for heating, a pipe is suitably arranged around the lower inner wall of the oven. It should have holes along its length at intervals of about 1 in. They should be of a size and so directed that the issuing flames do not give rise to highly

* Johns-Manville, 20 East 40th Street, New York, N. Y.

localized temperatures in the apparatus to be baked out. A small vent hole in the top of the oven will allow unburned gases to escape.

With electrical heating, commercially available heater strips or rods may be mounted on the inner walls of the oven. In some cases it may be more desirable to provide these inner walls with heaters of iron or nichrome wire either in coils or in straight lengths.

The more heavily insulated ovens require more heating and cooling time, but at operating temperature may require slightly less power than the lighter oven with a single polished metal wall. This latter oven, which may be conveniently formed of half-hard aluminum, has remarkably short heating and cooling times.

A very convenient oven to have available around a laboratory for baking out traps or lengths of tubing or for any general baking is formed by using a piece of rolled sheet iron such as a length of stove pipe of suitable diameter and length. First a layer or two of asbestos paper or tape is used to cover the metal pipe. Then iron or nichrome wire, say 0.020 or 0.030 in. in diameter, is wound around the pipe from one end to the other. The turns are ½ to 1 in. apart, and every three or four turns there should be a twisted loop an inch or so long which can be used as an adjusting "tap" during operation. Finally more asbestos paper or cloth tape is wound on to a thickness of ½ to ¾ in.

Such an oven has great possibilities of adjustment because of all the taps and the possibility of operating two halves in parallel. If the temperatures achieved are not as desired, it is no great task to rewire the device or rearrange the insulation.

If it is impossible to heat the entire evacuated apparatus because of the presence of waxes or rubber in the assembly, it may be possible to use a flame or partially enclosing electric heater to heat portions of the system.

Another method useful in removing occluded gases is to scour the chamber by means of a violent gas discharge. It may be desirable to introduce some preferred gas for this purpose, or it may be accomplished by the residual gases at a pressure of a few mm Hg.

Considerable gas can be driven from the glass walls of a vacuum system with a hand spark coil. This device, also used in leak detecting as discussed in the next chapter, is played back and forth over the glass surface. Except under the best vacuum conditions (better than 10^{-5} mm Hg) the localized high-frequency fields generated by the device are sufficient to cause ionization and bombardment of the walls with the resultant release of gas. This effect, although probably quite similar in action to the gas discharge scouring just described, is a much more localized process and occurs even though insufficient gas is present to maintain a glow discharge. In some cases of contaminated glass, for example, in the exhaust line near the oil diffusion pump where oil or oil vapor may be present, the use of the spark coil may give rise to decomposition and the deposition of a stable residue on the glass.

Electrodes in vacuum systems may be brought to high temperature by ion or electron bombardment, provided the arrangement gives convenient sources of ions or electrons together with accelerating means. Any electrode, or in fact any element in the vacuum system, which is to be operated at elevated temperatures should be previously outgassed at some higher temperature. Likewise any elements subjected to electronic or ionic bombardment should receive a more severe treatment of this kind before operation, since such bombardment is a very prolific source of gas.

In processing devices such as electronic vacuum tubes which contain metal parts and are ultimately to be sealed off it has become standard practice as part of the exhaust procedure to "bombard" the metal parts. By bombarding is meant that the parts are heated, usually to bright red, by means of induction from an externally arranged coil. This bombarding is usually started cautiously to avoid the violent gas discharge which would be set up by the high-frequency fields in case the pressure of the released gas is allowed to rise. After the initial large quantities of gas have been pumped away, the heated parts may be held up to temperature for any desired length of time to accomplish the outgassing.

Care should be taken that the coil not come into contact with the glass wall of the evacuated tube or glass may be cracked

from locally induced strains. Likewise care should be taken that the operator not come into contact with the coil or its leads to prevent deep slow-healing burns. The coils, which normally become quite hot in operation, are customarily made of copper tubing. They are cooled by water flowing through them.

The most efficient induction heating occurs when the heated part is centrally located in a simple solenoid or some variation of it. However, this often necessitates preplacing a coil before assembling the system or otherwise restricting the freedom of operation. Many shapes and arrangements of coils have been worked out to allow the induction field to be conveniently applied where desired. When conditions make it impossible to lower a coil over the work to provide uniform heating of concentric cylinders or disks, hinged double coils having specially formed coil shapes and resembling large tongs may be applied. Flat-wound "pancake" coils are often useful for parts such as "getter flags" and deflection plates, which present a broadside surface to the tube wall.

High-frequency converters (bombarders) of several designs are available in sizes varying from 1 or 2 kw up. A 4-kw or 5-kw unit will be found ample for nearly all experimental electronic work whereas for consistently small parts the 2-kw size may be sufficient. Interchangeable load coils of various shapes may be purchased or in special cases may be made up as desired. Some skill is necessary to bend copper tubing properly and to fit it with connections for water cooling. However, for short-time bombarding where the cooling may be dispensed with, satisfactory coils may be made of very heavy copper wire or rod.

9·6 Introduction of Gas into the Vacuum System

When a controlled quantity of gas at some particular pressure is desired in the vacuum system, the condition may be obtained by balancing the inflow of such gas against the pumping capacity in such a way that the desired pressure is obtained. Another method is to admit gas at a higher pressure than is desired and then pump the system down until the gauge indicates that the required conditions have been reached, at which time the pumps are valved off. The former method is better, particularly for

very low pressures, since the contamination due to inevitable residual gases will be more constant by that method.

Unless the gas is generated in the immediate vicinity, it will usually be at hand in glass bottles at low pressure or in high-pressure steel cylinders. It is unadvisable to attempt to make direct connection between a high-pressure cylinder and the vacuum system since the controls are very unstable when the pressure is reduced to the extent necessary to work into a vacuum system.

A number of methods may be used to introduce the small amount of gas required. One of the most simple and convenient

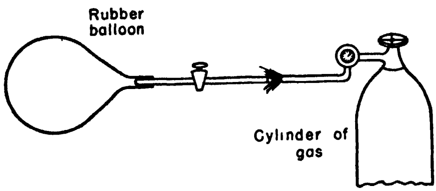


Fig. 9·4 Use of Rubber Balloon as Auxiliary Gas Container.

is an ordinary rubber balloon (Fig. 9·4). This balloon is inflated from the high-pressure cylinder through a reducing valve or by "cracking" the main valve or by holding the tubing nearly, but not quite, flush with the high-pressure outlet, but allowing some of the high-pressure gas to be lost through the "poor fit." The balloon should be filled and deflated once or twice for cleaning purposes and then filled for use as a gas container. It is usually attached to a piece of glass tubing provided with a stopcock. Thus, after being filled with gas, it may be connected to a vacuum system and will either serve to supply a slow leak or suffice for several fillings of the entire vacuum system.

Glass bottles, usually about 1 liter in capacity, are customarily used for supplying such high-purity gas as neon, helium, and xenon. They are available in either hard or soft glass and are provided with a small, finely drawn constriction or seal which is protected by the neck of the bottle. The gas is usually at about atmospheric pressure. When such a bottle is to be used it should be provided with two stopcocks sealed to the bottleneck (Fig. 9·5). Between the stopcocks and the bottle

should be placed a small slug of metal, say a short length of iron rod, which may be sealed in a small glass capsule when extreme purity is desired. This iron slug is used to break the small seal-off tip after the air in the bottleneck has been exhausted through the stopcocks.

Such an arrangement must be handled with care so that the iron slug does not break the tip before the air has been removed from the neck. The use of two stopcocks in series is recommended, first, to give a double seal against contamination of the

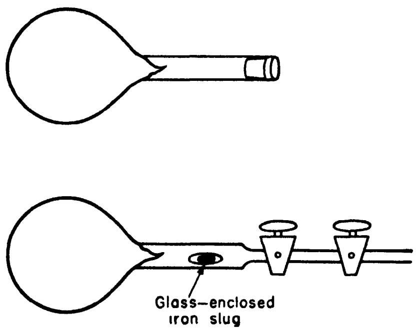


FIG. 9·5 Arrangement for Using Bottled Gas.

gas by leakage through the stopcocks and, second, so that the portion of tubing between the stopcocks may be used as a sort of dosing or gas-dispensing chamber. Thus after this space has been pumped out, the outer valve may be closed and the inner one opened and then closed again. The gas thus caught between the two stopcocks may be used in the experimental work and the main bottle of gas is not jeopardized.

The high rate of diffusion of hydrogen through palladium may be made use of in admitting this gas to a vacuum system. Figure 9·6 shows a convenient apparatus for this purpose. The metal-to-glass seal is made to soft glass. The long thin-walled palladium tube, closed at one end, is arranged so that one surface is exposed to the vacuum and the other to hydrogen atmosphere. The palladium tube, only a few mils in wall thickness and several inches long, is placed in series in an electrical circuit by collars around its two ends. The temperature may be

varied by the passage of current through it. The rate of passage of hydrogen may be controlled by adjusting the palladium temperature, the rate of diffusion increasing extremely at high temperatures.

If only a small amount of gas is needed in a system it may oftentimes be obtained by merely outgassing a wire containing that gas, which may be heated by the passage of electrical current through it. By this means, hydrogen in small quantities may be obtained from platinum or palladium wires and oxygen

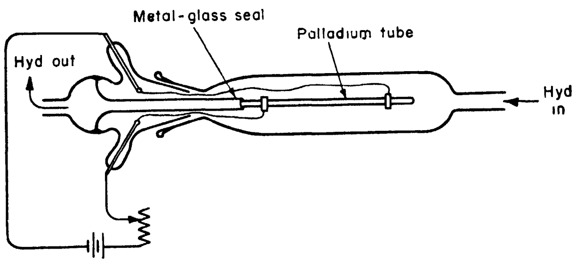


FIG. 9·6 Palladium Tube Arranged for Hydrogen Diffusion.

may be obtained from silver. Oxygen may also be introduced into a system by heating red mercury oxide in a convenient side tube.

9·7 The Manipulation of Cesium, Sodium, Mercury, and Similar Materials in the Vacuum System

The general method of moving such materials around in the vacuum system consists of distilling them from one position to another. Excessive temperatures are not required to give them fairly high vapor pressures. Likewise, they may readily be condensed by being brought in contact with areas cooled only to room temperature.

The first step is to get a suitable amount of the material from its normal storage condition into a side tube on the vacuum system. A minimum of contamination should accompany the material. As an example, sodium usually comes immersed in some protective medium such as kerosene. With forceps or tongs a small piece is placed in a glass container, which is flushed a num-

ber of times with a degreaser such as benzene or ether (carbon tetrachloride or chloroform should not be used). The sodium is then placed as quickly as possible through an open-side tube into an appendix type container sealed to the vacuum system.

Also sealed to the system in a convenient location with reference to the appendix another side tube should be placed with a number of smaller capsule-like side tubes fastened to it by means of constrictions. This arrangement is shown in Fig. 9·7. When the system is evacuated the appendix containing the sodium is

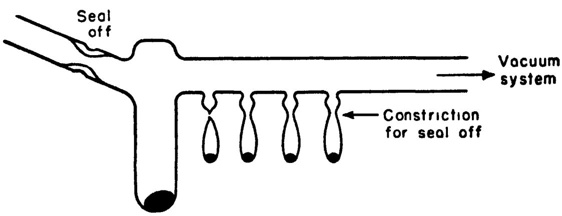


FIG. 9·7 Distillation of Metal into Ampoules.

heated slowly. The hand torch is convenient for this purpose as well as for the rest of the process. As the temperature is raised the sodium finally commences to leave the original piece and the vapor condenses on the cooler walls farther along the system. Excess heating should be avoided in all stages of such a process as the violent distillation which results tends to cause impurities to be carried along with the sodium metal.

From here on a technique must be followed of "herding" the vapor out of the original side tube over into the individual ampoules. This is accomplished by trying, in general, to keep all parts of the glass system warm except the ampoules and by torching toward the sodium from the direction opposite that in which it is to move. Since the sodium tends to creep along the walls, it is often convenient to segregate a small amount and drive it along the glass to its destination and then return with the torch to the original supply for another allotment.

If considerable impurity is carried over from the first side tube it may be found desirable to distill the sodium into several intermediate tubes on the way to the ampoules. Once an ampoule has received its allotted share (it should not be filled more than

a third full), a small pointed flame is played around the constriction until the glass shrinks together and the capsule can be sealed off and pulled away.

Later it may be introduced into the experiment. One method is by scratching it with the glass knife and then breaking it under vacuum in a rubber tube which is attached to the vacuum system as shown in Fig. 9·8. This should be arranged in such a way that after the breakage the capsule parts may be shaken down into a glass chamber, the tube leading to the rubber hose

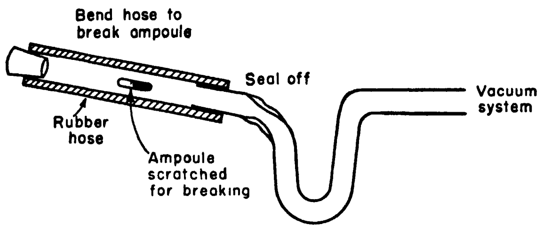


FIG. 9·8 Introducing Metal into Vacuum System by Breaking Ampoule.

sealed off, and the sodium then driven to the desired position by flaming as described before.*

This same method with suitable variations may be followed for all materials of this nature. Each has its peculiar characteristics and actions when flamed under vacuum. The degree of vacuum required during distillation depends upon the material and upon the purity desired in the final product. While the best of vacua are necessary for such materials as cesium and barium, ordinary forepump pressures may suffice for mercury.

As a matter of fact, distillation is one of the best methods of cleaning mercury. Figure 9·9 shows an apparatus for such work. To save undue contamination of the still it is best to preclean the mercury. This may be done by agitating it in conveniently small portions in a wide-mouth bottle of dilute nitric acid.

* Sodium, potassium, and cesium must be handled very carefully to avoid sudden fires. They react violently when in contact with water, and severe chemical burns may result from the spattering of the material which occurs. The operator should wear suitable protection.

Different materials may be acquired in various forms and under different conditions of storage, and each must be handled in a suitable manner. In some instances the material may be so extensively used commercially that it is available in rather convenient form. For instance, the "getters" used in the radio tube industry contain active metals in the form of pellets or enclosed in hollow wires which may be "flashed" by heating when placed in the vacuum. Among the metals thus available are barium,

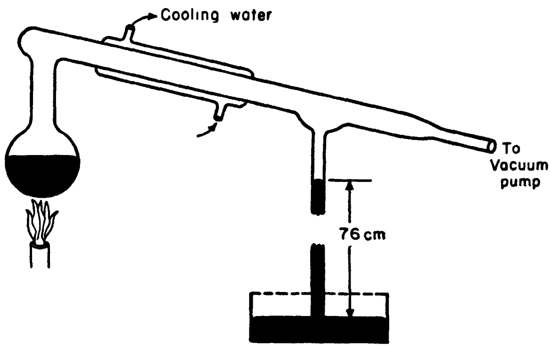


FIG. 9·9 Mercury Purifying Still.

strontium, magnesium, calcium, and aluminum, alone or in various combinations. In such cases the necessity for distilling the active metals into ampoules for dosage is eliminated as both the amount of material and its point of application are easily controlled.

If a pellet is to be used it is usually mounted in a small cup-shaped indentation on a piece of metal such as nickel. A piece of fine wire mesh placed over it and spot-welded around the edges will serve to retain it in the cup. When the active metal is used as a core in wire a piece of the wire is mounted in a similar fashion on a metal flag. As supplied, one side of the wire is customarily thinned to allow easier escape of the metal.

This metal flag is oriented in such a way that when it is heated by means of a high-frequency bombarder coil to the temperature at which evaporation of the active metal occurs, the material will deposit on the desired surface. To avoid contamination by the

gases emitted from the flag as it is heated, it is desirable to raise the temperature several times to just under that required for evaporation. Final evaporation should then be carried out after the gases thus liberated have been pumped away.

A technique similar to the above may be used for some materials which are fairly stable when exposed to the atmosphere. For instance, the iodides, chlorides, or bromides of sodium or potassium, among others, may be deposited in vacuum on prepared surfaces from small pans or cups containing the material and heated either by induction or by auxiliary heater coils.

9·8 Getters

Regardless of how thoroughly its components are cleaned and how well they are baked and degassed on exhaust and regardless also of the quality of vacuum reached during exhaust, the fact remains that a sealed-off tube tends to become "soft," that is, gassy, during life because of gases liberated during operation or because of diffusion from some of its parts. These gases include oxygen, nitrogen, carbon monoxide, water vapor, and chlorine. Often because of the effect of gas on its characteristics the tube is of little value unless it has a "hard" vacuum, and this liberation of gas gives rise to shortened tube life.

The procedure known as "gettering" makes it possible to extend tube life considerably. It consists of introducing materials into the tube which will react with the undesirable gases in such a way as to immobilize them. Such getter action can be remarkably efficient. In the manufacture of many radio tubes it is standard practice to seal the tubes off at comparatively poor vacua—well above a micron—and depend upon getter action to reduce the tube pressure to values often better than 10^{-5} mm Hg.

The list of materials used as getters is extensive, including barium, strontium, magnesium, thorium, aluminum, tantalum, zirconium, uranium, and misch metal (largely lanthanum and cerium). It would seem that by virtue of their function, the trapping of gas and vapors, many of the materials discussed in Chapter 3 should be included here, but the term "getter" usually is reserved for a material such as one of those in the above list which is dispersed in a sealed-off tube or lamp. Some of these

materials are highly selective in their functioning. The inert gases are, of course, unaffected by them. In many cases where the operation of a tube depends upon the purity of an inert gas filling, getters make it possible to maintain that purity by their removal of other gases evolved during operation.

To facilitate handling of the active gettering materials during the construction of a tube they are available in a number of different forms protected either chemically by the stable nature of the compound or physically by being coated with a protecting layer of some sort.

Barium is the most commonly used material. It may be had combined with strontium, magnesium, or aluminum in one form or another. In some cases the materials are alloyed and in pellet form; in others they are enclosed as cores in iron, copper, or aluminum wire.* In the Batalum † getter the carbonates of barium and strontium are coated on tiny coils of tantalum or in tiny V-shaped troughs of tantalum ribbon.

In every case the amount of getter is under close control and is released in the tube by the application of heat. The pellets and wire may be mounted on "getter flags" as described in the last section and heated by high-frequency induction. Wire or ribbon-like, the Batalum getter may be connected across two leads in the stem and heated by the passage of current through them, or the ends may be electrically connected in a closed loop in which the necessary heating current may be generated by the induction coil.

Release of the getter material by this means is called "flashing" or "exploding" the getter. This process is essentially one of evaporation, and the material proceeds outward in straight lines until it is intercepted by a surface upon which it can condense. The surface must be sufficiently cool. The getter flash by means of the flag or other shielding should be directed to some part of the tube where its presence can cause no irregularity in tube operation. If the vacuum is good and the surface smooth the result will be a smooth metallic coating of active metal. If the surface is not smooth the coating will not have a metallic

* King Laboratories, Inc., 205 Oneida Street, Syracuse, N. Y. Kemet Laboratories, Inc., West 117th St. and Madison Ave., Cleveland, Ohio.

† Radio Corporation of America, RCA Victor Division, Harrison, N. J.

sheen. In the presence of inert gas the deposited layer will be a diffuse spongy non-metallic-looking coating. If active gas is present the appearance will depend upon the gas, and may be anything from gray-white to dark brown.

If the getter material can be deposited in a diffuse rather than a shiny metallic layer its gettering efficiency is greatly increased. In one method of doing this * inert gas at a few millimeters pressure is introduced while the getter is flashed. This results in the desired type of deposit, and the inert gas is subsequently removed.

Ehrke and Slack[3] found barium and misch metal to be the most active getter materials. However, barium becomes less effective in the presence of mercury vapor. Misch metal is somewhat less affected by mercury vapor whereas magnesium and thorium in the presence of mercury pick up oxygen but not hydrogen.

Zirconium and tantalum function as getters if operated at elevated temperatures. Zirconium is used in the ductyl form as wire or ribbon or in the powdered form sprayed in a suitable binder on an electrode, usually of molybdenum, which may be operated at a suitable temperature when the gettering action is desired. Apparently there is some difference of opinion concerning the proper gettering temperatures. According to Fast,[4] gettering should be accomplished with one piece of the metal held between 300° and 400°C to take up the hydrogen and another piece maintained above 1000°C to remove oxygen and nitrogen. Other investigators[3, 5] have found somewhat different effective temperature ranges.

Tantalum is another effective getter when hot. It is very effective at around 600° to 800°C, but re-emits the gas at higher temperatures. In fact the ease with which it is outgassed at higher temperatures is a factor in its choice as an anode in some power type radio tubes.

9·9 Determination of Pressure of Sealed-Off Tubes

Many times it is desirable to know the pressure of a sealed-off tube containing gas. In some cases this may be anticipated

* Philips Company's British Patent 471409.

in its construction by including in the tube some means of measurement, such as the elements necessary for an ionization gauge or some other electrical gauge or other device. Some radio tubes and cathode ray tubes have sufficient electrodes to serve as ionization gauge elements. In other tubes a vibration quartz fiber may be included such as was discussed in the chapter on gauges.

At other times, however, no such arrangement is available, and the method of allowing the volume of sealed-off gas to expand into another volume may be used. In this case a pressure gauge is provided on the receiving chamber so that both its original

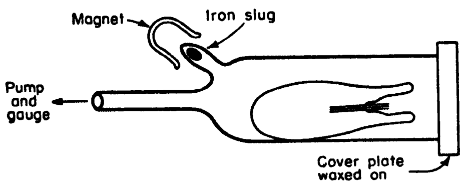


Fig. 9·10 Breaking Gas Bulb in Known Volume.

pressure and the pressure P_2 after the expansion may be measured. The volume V_2 of the system into which the gas is expanded must be determined, and after the operation is complete, the volume V_1 of the measured tube must be obtained. Then, from the relation $P_1V_1 = P_2(V_2 + V_1)$ the pressure P_1 in the original container may be calculated. The receiving chamber should be of such a size that the pressure after expansion will be an easily and accurately measured value.

If convenient, the tube under investigation may be placed completely inside the receiving chamber and broken by means of an iron slug held in readiness by an external magnet as shown in Fig. 9·10. Many times the tube to be studied is enclosed in an envelope of the kind standard for radio tubes and lamp bulbs. The evacuation of these envelopes is carried on through a very small glass tube, opening into the envelope near the press of the stem (Fig. 9·11). This tube extends downward from the stem, and in manufacture the tube is sealed off by shrinking the tubulation with a flame until the hole closes up and the glass becomes so soft that the entire tube may be drawn away, thus breaking its vacuum connection. This tubulation, which ordi-

narily is protected by the tube base, becomes very convenient in this expansion method of measuring the gas content of an envelope. The base is flamed carefully until the basing cement breaks down, after which the base may be pulled away from the

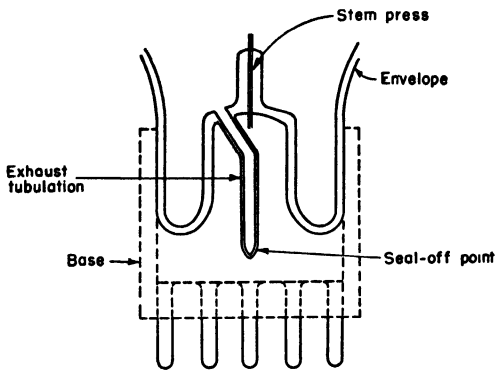


FIG. 9·11 Arrangement of Tubulation in Tube Base.

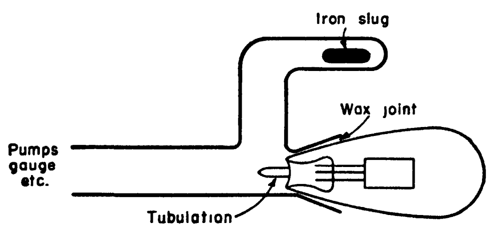


FIG. 9·12 Expansion of Unknown Gas into Known Volume by Snapping Tubulation.

tube. The envelope may be waxed or otherwise attached to the vacuum system in such a way that by external means the tubulation may be rapped sharply and snapped off. Figure 9·12 shows such an arrangement.

9·10 Sputtering

The disintegration of an electrode under positive ion bombardment has long been experienced in electron devices. Although occurring more commonly in the presence of gas at low pressures, its presence is felt also in such supposedly high vacuum tubes

as cathode ray tubes, where the process may be responsible for removal of the cathode coating and may sometimes even drill holes in the metal cathode cap.

The phenomenon known as "sputtering," by which a bombarding positive ion can cause the bombarded cathode material to be literally blasted out and deposited elsewhere, is not understood thoroughly. A number of theories have been advanced, but none has proved to be completely acceptable. However, the following facts have been established:

1. The particles leaving the cathode may be either negatively charged or uncharged.
2. The particles may be single atoms or clusters of atoms.
3. With the exception of hydrogen, which is one of the best gases for sputtering, the effect is more pronounced the heavier the gas.
4. There appears to be no consistent relationship between any physical characteristic of metals and their resistance to sputtering.
5. For given physical conditions the rate of sputtering increases with both the ion accelerating potential and the current.

The relative rates of sputtering of metals in some gases is shown in Table II.

Although the effects of sputtering are usually highly undesirable as encountered in electronic devices, the phenomenon can be made useful. Under proper conditions it is a very useful means of depositing metallic films on glass, ceramics, or other metals. The conditions for successful sputtering are not at all critical. A typical bell jar arrangement for the process is shown in Fig. 9·13.

The gas pressure is usually controlled by balancing the inlet against the pumps, using a controlled inlet leak alone or combining this with a valving action such as a partially open stopcock in the pump line. The actual pressure, which may be anything from one or two mm Hg down to a few microns, depends upon the other conditions and is adjusted as described later. The most commonly used gases are hydrogen, argon, and air. Air is fairly satisfactory for sputtering materials which do not

oxidize easily. Argon, usually accepted as the best gas for sputtering, is not sufficiently superior to hydrogen to offset its expense, with the result that hydrogen is usually used.

TABLE II

Sputtering Rates of Metals *

Observer	Gas	Rate of Sputtering in Descending Order
Crookes	Air	Pd, Au, Ag, Pb, Sn, Pt, Cu, Cd, Ni, Ir, Fe, Al, Mg
Kohlschütter	N_2	Ag, Au, Pt, Pd, Cu, Ni
Blechschmidt	A	Cd, Ag, Pb, Au, Sb, Sn, Bi, Cu, Pt, Ni, Fe, W, Zn, Si, Al, Mg
Güntherschulze	H_2†	Bi 1470, Te 1200, As 1100, Tl 1080, Sb 890, Ag 740, Au 460, Pb 400, Zn 340, Cu 300, C 262, Sn 196, Fe 68, Ni 65, W 57, Co 56, Mo 56, Mn 38, Cd 32, Al 29, Cr 27, Ta 16, Mg 9
Güntherschulze	O_2†	Zn 1030, Tl 650, Ag 614, Au 423, Pb 320, Cu 236, Sn 227, Fe 86, Mo 80, W 49, Ni 52, Cd 28

Sources:

Sir W. Crookes, *Proc. Roy. Soc.* (A), Vol. 50, p. 88, 1891.

V. Kohlschütter, *Zeits. f. Elektrochem.*, Vol. 15, p. 316, 1909. *Jahrb. Radioaktivitat*, Vol. 9, p. 335, 1912.

A. Güntherschulze, *Zeits. Physik*, Vol. 36, p. 563, 1926; Vol. 38, p. 575, 1926.

* From John Strong, *Procedures in Experimental Physics*. Copyright, 1938, by Prentice-Hall, Inc., New York. Reprinted by permission of the publishers.

† Numbers give rate of sputtering in milligrams per ampere hour under conditions of cathode fall of 770 volts and current density of about 7 ma per sq. cm.

The discharge may be excited by either alternating or direct current. When accurate control is necessary, a d-c source which is subject to controlled variation and measurement is desirable. However, an ordinary high-voltage transformer gives very good results. The type used in sign lighting is convenient. In either case potentials of 1000 volts or more are required—5000 or 10,000 volts being adequate.

The cathode usually consists of a sheet or plate of the metal to be deposited, although if the piece receiving the sputtered

layer presents a surface other than flat, it may be desirable to match the cathode shape to it. Normally the cathode is suspended as shown in Fig. 9·13 since the object being sputtered may then be easily supported upon a simple table or frame below it.

The supporting plate must be conducting since it serves as the anode, but it should be a material having a low sputtering

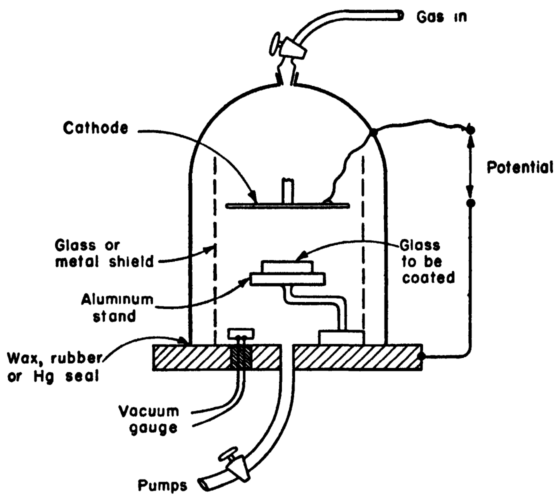


FIG. 9·13 Bell Jar Apparatus for Sputtering.

rate, particularly if the discharge is activated by alternating current. Aluminum serves admirably in this application. It is desirable to shield all wires in glass tubing, and in general every attempt should be made to confine the discharge to the region between the cathode and the anode plate. The separation of the electrodes can best be determined by experiment. To start with, some value from 5 to 10 cm is reasonable.

The general procedure to be followed in sputtering is quite simple. The parts are assembled, care being taken to insure extreme cleanliness of all parts and particularly the piece to be sputtered. The pump should be allowed to pull its best vacuum before the gas is admitted. The voltage is then turned on (negative to the cathode if direct current is used) and the gas is ad-

mitted slowly. The discharge will build up slowly as the pressure increases. The region between the electrodes should be watched closely. A background of dark cloth or paper may be of help in observing the discharge. The correct condition is obtained when the discharge in the inter electrode region shows a cathode dark space of at least one centimeter and preferably one which fills about half the space between electrodes.

An opaque coating of metal may be sputtered in from a few minutes to a few hours, depending upon experimental conditions. The distribution of the layer may or may not be uniform, and here again the cathode shape, electrode spacing, gas pressure, and nearly any other variable may be responsible. Although some success is obtainable with sputtering almost immediately, reproducible results with controlled uniformity and thickness are obtained only after considerable experience.

9·11 Evaporation of Metals

In an earlier section we described the method of moving various materials around in a vacuum system by means of distillation. That technique actually is one of evaporation, but those materials were characterized by requiring comparatively low temperatures and the procedure was thus classed as a separate technique.

The technique of evaporation as described in this section will consider those cases where comparatively high temperatures are required. Although the evaporation of the getter materials such as barium, strontium, magnesium, and aluminum were discussed in Section 9·7, as well as in Section 9·8, the techniques described here are also applicable to them.

In the evaporation process a much better vacuum is required than in sputtering since it is usually desired that the evaporated material travel in essentially straight lines from the evaporating source to the material being coated. This calls for vacua of the order of 10^{-4} mm Hg or better. The work is usually carried out in an apparatus similar to that shown in Fig. 9·14.

The apparatus consists essentially of a demountable bell jar with a suitable pumping system and gauges. A holder for the object to be coated is provided as is an evaporating source, usu-

ally either a cup-shaped furnace or a filament. Between the source and the object may be arranged a shutter activated magnetically or by other remote control means. Other shields may be arranged to prevent evaporating material from reaching areas such as the bell jar or other places requiring such protection. Liquid air is desirable when mercury pumps are used but may be dispensed with when oil pumps are used.

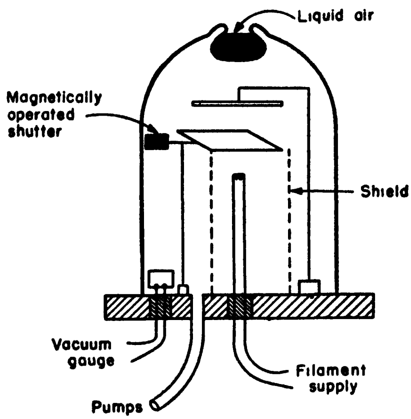


FIG. 9·14 Bell Jar Apparatus for Evaporation of Metals.

The type of evaporating sources most used are the simple helical coil of one or more turns, the close-wound conical basket, and the ceramic crucible. Figure 9·15 shows forms of them. The ceramic crucible may be made up by repeatedly coating a conical wire basket with a water paste of BeO or Al_2O_3 until it bridges from wire to wire and closes up, forming a crucible. The ceramic should be thoroughly baked before use preferably to a temperature as high as will be reached in operation. To avoid cracks such crucibles should always be brought to temperature very slowly and cooled the same way.

If a metal does not evaporate smoothly from the heater or refuses to adhere to the heater wire, it may help in some cases to plate the metal to the heater wire. It may also be advantageous to prefuse the metal to the heater wire by heating in a hydrogen or helium atmosphere.

A number of metals may be used in the heater coils. Tungsten, tantalum, molybdenum, iron, nickel, and Chromel have been used. The choice depends upon the temperature to be reached and the chemical reaction of the heater with the evaporated metal. Many attempts to evaporate aluminum from tungsten fail because the materials alloy and the heater burns out at this point. Strong [6] points out that tungsten has a limited solubility in aluminum (3 per cent by volume) and that aluminum may be satisfactorily evaporated if large tungsten wires are used, thus insuring strength of the heater coil after the aluminum has combined with its share of the tungsten. Olsen, Smith, and Crittenden [7] have found that to evaporate cobalt, nickel, and iron from tungsten, the amount of tungsten must exceed the evaporated metal by 3 to 1 in weight.

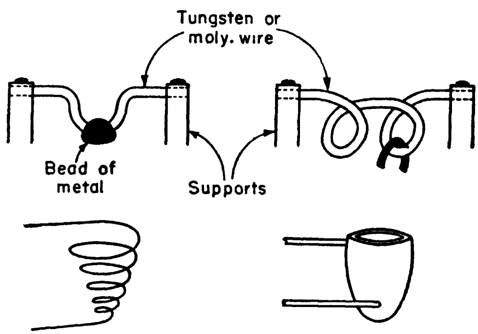


FIG. 9·15 Structures for Supporting Metals to Be Evaporated.

When the materials have been properly cleaned and adjusted in place the bell jar is evacuated. After the proper vacuum is reached the temperature of the heater is slowly increased. The shutter should be in position during this time as considerable agitation at the heater may occur which can throw heavy particles and impurities onto the prepared surface unless it is protected. As soon as the proper temperature is reached and stable conditions attained the shutter can be opened, allowing the metal to evaporate to the desired surface. Stable operation does not preclude all motion at the evaporator since bubbles of

the molten metal will constantly be changing their position and size.

When possible it is desirable to keep the work above the source to prevent heavy particles from dropping on it. It must also be remembered in arranging the work that the evaporated material leaving the source travels outward in all directions. In the case of large areas of work this will result in non-uniform deposition which may be obviated by arranging the work at a greater distance. In this case the deposited material arrives at the work along more nearly parallel lines.

It is sometimes possible to arrange a metallic crucible in such a position that it may be heated by high-frequency induction from a surrounding coil placed outside the vacuum.

A different method of heating is shown in Fig. 9·16. A small crucible of conducting material such as graphite, platinum, or tantalum is surrounded by a tungsten coil filament. Such a cup-shaped container is easily made by drilling into the end of a short piece of metal rod. The crucible is maintained at a suitable positive potential (a few hundred to a few thousand volts) with respect to the filament. When the filament is heated it emits electrons which bombard the crucible sufficiently to raise its temperature as desired.

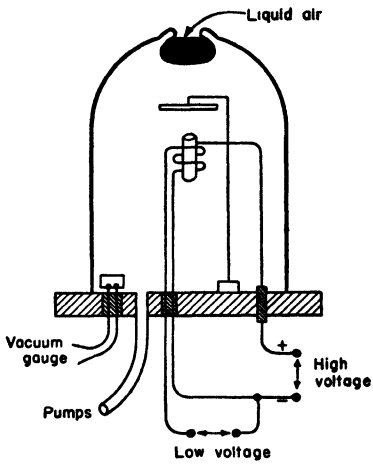


FIG. 9·16 Heating Crucible by Electron Bombardment.

The power dissipated as heat at the crucible is given by the product of the accelerating voltage times the bombarding current. Since the emission current used in bombarding is more likely to be measured in milliamperes than in amperes it is plain that high accelerating voltages will be required. Because of the probability of a gas discharge due to the electron emission and

the high accelerating voltage very good vacua must be maintained for this method of heating.

Table III indicates the temperatures at which metals will evaporate.

TABLE III

EVAPORATION TEMPERATURE * OF DIFFERENT METALS †

Material	Evaporation Temp. T° Absolute *	Material	Evaporation Temp. T° Absolute *
Hg	320	Pb	1000
Cs	433	Sn	1148
Rb	450	Cr	1190
K	480	Ag	1319
Cd	541	Au	1445
Na	565	Al	1461
Zn	623	Cu	1542
Mg	712	Fe	1694
Sr	811	Ni	1717
Li	821	Pt	2332
Ca	878	Mo	2755
Ba	905	C	2795
Bi	913	W	3505
Sb	973		

Sources:

E. Baur and R. Brunner, *Helv. chim. Acta.*, Vol. 17, p. 959, 1934.

W. Espe and M. Knoll, *Werkstoffkunde der Hochvakuumtechnik*, p. 358, Julius Springer, Berlin, 1936.

M. Knoll, F. Ollendorff, and E. Rompe, *Gasentladungs-Tabellen*, Julius Springer, Berlin, 1935.

Landolt-Bornstein, *Phys. Chem. Tabellen*, Fifth Ed., Julius Springer, Berlin, 1923–1936.

W. Leitgebel, *Metallwirtschaft*, Vol. 14, p. 267, 1935.

* Temperature at which vapor pressure equals 10^{-2} mm Hg.

† From John Strong, *Procedures in Experimental Physics*. Copyright, 1938, by Prentice-Hall, Inc., New York. Reprinted by permission of the publishers.

As a result of an investigation of a large number of metals Olsen, Smith, and Crittenden[7] suggest evaporation methods shown in Table IV.

TABLE IV

SUGGESTED METHODS FOR EVAPORATION OF VARIOUS METALS

Metal	Method for Small Quantities	Method for Large Quantities
Aluminum	Helical coil of W	Helical tungsten coil
Antimony	Conical baskets of W, Ni, or chromel	Alundum crucible
Arsenic	Alundum crucible	Alundum crucible
Barium	Conical baskets of W, Ni, Fe, or chromel	Conical baskets of W, Ni, Fe, or chromel
Beryllium	Conical basket of W	.....
Bismuth	Conical baskets of W, Fe, Ni, or chromel	Alundum Crucible
Calcium	Conical basket of W	Alundum crucible
Cadmium	Conical baskets of W, Ni, Fe, or chromel	Alundum crucible
Carbon	Unsuccessful	Unsuccessful
Chromium	Conical basket of W	..
Cobalt	Helical coil of W	Alundum or BeO crucible
Columbium	Helical coil of W	..
Copper	Helical coil of W or conical basket of W	Alundum crucible
Germanium	Conical basket of W	Alundum crucible
Indium	Conical baskets of W or Fe	Alundum crucible
Iron	Helical coil of W	Alundum or BeO crucible
Lead	Conical basket of Fe	Alundum crucible
Magnesium	Conical baskets of W, Ni, Fe, or chromel	Alundum crucible
Manganese	Conical basket of W	Conical basket of W or Alundum crucible
Molybdenum	Melting of molybdenum filament	......
Nickel	Helical coil of W	Alundum or BeO crucible
Palladium	Helical coil of W	.....
Phosphorus (red)	Unsuccessful	Unsuccessful
Platinum	Helical coil of W	....
Selenium	Conical baskets of W, Ni, Fe, or chromel	Alundum crucible
Silicon	BeO crucible	BeO crucible
Silver	Helical coil or conical basket of W	Alundum crucible
Strontium	Conical basket of W	Large conical basket of W
Tellurium	Conical baskets of W, Ni, Fe, or chromel	Alundum crucible
Thorium	Conical basket of W	Conical basket of W
Tin	Conical basket of W	Alundum crucible
Uranium	Conical basket of W	......
Vanadium	Conical basket of W	Conical basket of W
Zinc	Conical baskets of W, Ni, Fe, or chromel	Alundum crucible
Zirconium	Conical basket of W	......

From L. O. Olsen, C. S. Smith, E. C. Crittenden, Jr., *J. Applied Phys.*, Vol. 16, pp. 425–434, July 1945.

9·12 Surface Coatings

This section is a discussion of methods of applying conducting materials to surfaces by means other than sputtering and evaporation, which were recently described. For the most part, these coatings are for the purpose of making glass conducting at specified regions on the inside surface of the vacuum chamber or its elements. On the other hand, the purpose may be to provide a surface with a desired optical characteristic. Possibly it must be opaque or reflecting or non-reflecting. Some of the more common materials used are silver, platinum, gold, aluminum, and graphite. Ordinary silver plating is omitted here because it is adequately treated in many handbooks and textbooks and actually it is not used extensively in the preparation of electronic apparatus.

Silver is usually applied from a paste, available commercially, which may be applied with a brush. The thickness is quite easy to control, either by applying thickly at the start or in successive layers. The coated glass parts are heated in air to several hundred degrees centigrade to drive off the volatile carrier. The result should be a white coating sometimes having a bronze tinge. Thorough polishing brings forth the metallic silver appearance.

Platinum and gold are available as salts in solution in lavender oil. The solution is painted on the desired area and the piece is either baked or flamed in much the same manner as was described in the case of silver paste. Both these metals will take on their characteristic surface appearance immediately after heating. Heavier coatings are obtained by repeating the operation as desired.

Graphite is available in colloidal form * as a thick paste. By carefully following the manufacturer's instructions it may be diluted so that it may be painted or flowed on where desired. It is very necessary in the case of Aquadag that the manufacturer's instructions be followed explicitly. If not, the graphite may go out of suspension in such a way that it will "clump" and refuse to "wet" or spread uniformly over the surface of the glass.

Conducting aluminum coatings may be painted on glass. The

* Aquadag from Acheson Colloids Corp., Port Huron, Mich. Dixonac from Dixon Crucible Co., Jersey City, N. J.

aluminum powder is first carefully degreased and then stirred into a thin collodion solution. This is then painted on the glass and heated in air to a temperature just above that required to remove the collodion. If the temperature is too high the aluminum will not have good conducting properties.

Leakage across internal insulators in electronic devices has always been a problem. Manufacturers of radio tubes have developed various coatings to minimize this difficulty. One of the more common methods is to paint the surface with milk of magnesia.

A technique quite successful in minimizing leakage across external insulators such as high-voltage bushings makes use of Dri-Film.* The area to be treated is exposed to the Dri-Film vapors and then baked for a few minutes. When a surface prepared properly by this method is exposed to moisture, the moisture will collect in small isolated droplets and minimize surface leakage.

In all the coating techniques discussed above, the first requisite is an extremely clean surface. All the techniques yield disappointing results at times for no apparent reason. Sometimes the failure may be traced to the cleaning, sometimes to impurities in the material which is being applied, and sometimes to the wrong baking-out procedure.

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* Dri-Film from Chemical Division, General Electric Co., Schenectady, N. Y.

Chapter 10

LEAK DETECTION

10·1 General Discussion

The term "leak detection" actually is twofold in its implication. First, leak detection may mean detection of the fact that the system contains a leak and, second, detection of its whereabouts. It might seem that the realization that a system contains a leak would be quite obvious, but actually there are many times when it is nearly impossible to be certain. Because of this uncertainty, we talk of "virtual" leaks and "true" leaks.

Under the section on gauges, we saw that the pressure in a system was indicated to us by means of various phenomena such as ionization, the color of a gas discharge, the rate of cooling of a sensitive member, etc. Now, in general, these devices tell us something of the amount of gas present in a system, but they do not always give us the true picture of the kind of gas or where it comes from. For instance, a poor reading on a vacuum gauge may be due to an actual air leak through the chamber wall (a "true" leak) or it may be due to the evolution of gas from the inside surface of some of the apparatus (a "virtual" leak) or it may be due to the inability of the pumping system to attain a lower vacuum. With any pumping system there is back diffusion of the pump vapors, that is, mercury or oil; and they eventually find their way throughout the system and may affect the gauge readings.

When the anticipated vacuum is not realized in a vacuum system it is desirable first to determine the reason before attempting the cure. When it is possible to excite the gas with a spark coil and view its coloring, a little experience will quite often enable one to judge whether a true air leak is present. The glow discharge of such an air leak has a very pronounced red com-

ponent. On the other hand, oil vapors and the gas present from contamination usually have the more somber hue of blue-gray.

If the system can be sealed off from the pumps by means of a stopcock or valve, the gas, either by evolution or through the true leak, will tend to build up a higher pressure, which at times will make its identification more certain.

True leaks usually build up to these higher pressures at a greater rate than the residual surface gases and the gases evolved from foreign material, since with the latter an equilibrium pressure is reached which retards further evolution.

Another indication that the inability to reach the desired ultimate vacuum is due to other than a true leak may be made evident by the application of heat to parts of the vacuum system or by the application of a scouring "glow discharge" as described earlier. If these treatments give rise to a large increase in gas pressure each time they are applied, it is quite probable that the trouble may be due to an excessive amount of gas adsorbed on the surfaces in the system.

The easiest way to determine the source of gas in a simplified system may be to go through the motions of searching for an actual leak. A good general rule to follow in assembling complicated vacuum systems is that each component of the system should be tested individually. Then, as each piece is added, its effect on the vacuum is established before addition of the next element. Some of the techniques used in leak searching are described here.

10·2 The Use of a Spark Coil

We have already discussed a spark coil as a means of indicating the pressure of gas in a vacuum system and have pointed out that from the colored gas discharge considerable information may be obtained as to the kind of gas present. The color of the discharge depends upon the kind of gas being excited, and we can make use of this phenomenon by making it possible for a specified foreign gas to enter the vacuum system through the leak. For instance, if the discharge is showing the typical red air color and the suspected region is flooded with ether by means of an atomizer, a brush, or a cloth saturated in it, the ether will

enter the vacuum system through the leak and the discharge color will suddenly change.

If the leak is bad the color will drop to a barely imperceptible gray; if the leak is smaller the strength of the discharge stays about the same, but the color takes on a bluer or a blue-gray tint. The best practice usually is to cover large portions of the vacuum chamber until an indication is encountered, after which the point of application may be more and more localized until the exact point of leakage is known.

Instead of ether a jet of hydrogen is often used in leak searching. When neither can be used carbon tetrachloride may be substituted. It will not pass through extremely small holes as well as the ether or hydrogen but it is safer. It is obvious that extreme care is necessary for hydrogen or ether and particularly so when a spark coil is used. The main ether supply should be kept at a distance, and only a small amount kept in the container used. Adequate ventilation should be maintained to insure against explosions as well as the breathing of the organic vapors.

In glass systems pinholes are often encountered, usually where a glass seal has been made. When the tip of the spark coil is passed over the glass and such a pinhole is encountered the area lights up to incandescence very much like a small steady localized spark. Care must be taken in the use of certain types of spark coils that the tip is not held too long in one place on the glass or a small hole may be actually "drilled" through the wall. In a region of very thin glass, it may be impossible to bring the coil up to the wall without puncturing such a hole, and many times this will lead one to "find" leaks which actually are not present.

10·3 The Use of Gauges in Leak Detection

Many of the gauges described previously may be used in the search for true leaks in a vacuum system. The procedure in general is very much like that described in the use of the spark coil. A gauge will be of little value unless the pressure in the system falls within the sensitive range of the gauge. Thus Pirani gauges and thermocouple gauges are of more value for pressures above a micron, whereas ionization gauges become very useful only below this value.

Gauges which are not continuously indicating, such as the McLeod, are of little use in leak detection since it is necessary to have an indication which will respond quickly to changes in the vacuum condition.

When the Pirani or thermocouple gauges are used the suspected regions are exposed to the vapors of ether, carbon tetrachloride, or a jet of hydrogen or other gas as was described in the section concerning the spark coil. When the leak has been found, the hydrogen will diffuse through the small hole much faster than the molecules of air. The gauge will indicate an increase in pressure due in part to the slightly increased pressure and also to the different gauge characteristic for the new gas. The smaller the rate of leakage, relative to the rate of pumping, the more difficult it will be to determine a leak by this method since only very small deviations on the gauge will result.

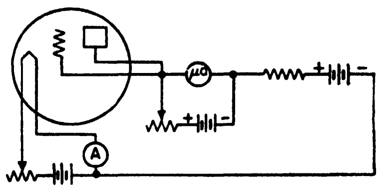


FIG. 10·1 Bucking Circuit Using Ionization Gauge for Leak Detection.

The ionization gauge is an extremely sensitive device for use in leak detection. This is particularly so if the effect of contaminating gas on the emission from the tungsten filament is utilized. With the simple gauge circuit in which the filament emission is not stabilized it will be found that the admission of different gases through a leak will cause varying phenomena.[1, 2] The ion current usually will indicate pressure changes in the conventional manner. However, depending upon the type of gas used, the tungsten filament will either increase or decrease markedly in its emission characteristic. This change in emission may be more sensitive an indicator than the variation in the ion current, and with a few changes in the circuit this phenomenon may be used as a leak detector having many times the sensitivity of the gauge as normally used. Figure 10·1 shows a circuit suitable for the application. The principle is essentially that of bucking the emission current to give a balanced reading of the indicating instrument (the microammeter in Fig. 10·1). Slight deviations in the emission are readily noted. One of the draw-

backs to leak hunting with gauges is that one person is required to watch the gauge constantly. This has been obviated[3, 4] by schemes where current fluctuations are converted to audio signals.

10·4 The Use of Applied Pressure in Leak Detection

Many times it is possible to locate leaks, particularly of the larger size, by applying pressure internally to the chamber. The leak may be made evident by submerging the chamber in water and looking for air bubbles, or a soap solution may be applied over the surface and the leak will be made known by the appearance of bubbles. This technique is not advocated for extremely small leaks as it is not sufficiently sensitive. Also it should be used with some caution in vacuum chambers fitted with gasket joints since many times the closure of such a joint requires higher pressure on the outside, and a reversal of this arrangement may merely open the system to relieve the pressure.

10·5 Leak Detection by Systematic Coating of the Chamber

In a method which is very satisfactory, but entails considerable work, a sealing material such as lacquer, glyptal, or some other suitable vacuum cement is applied systematically over the suspected regions in the system. The vacuum gauge is observed closely during this process, and as a leak is covered its position is indicated by an improvement in the vacuum gauge reading.

The coating process should not be carried on too quickly or there may be considerable inaccuracy in the determination of the position of the leak. It must be remembered that there may be a slight time lag with this method to allow removal of the volatile components in the coating material and allow it to harden sufficiently.

This scheme is particularly valuable when there are a large number of leaks in the chamber. In such instances the other methods may not give an indication because the local application of ether to one hole may be completely overshadowed by the effect of all the other leaks. In other words, the air entering from all the leaks is diluted only slightly by the ether entering through the one hole and the usual leak indication is masked. The method of systematic coating obviates this difficulty.

However, unless it is possible to leave the material on the vacuum chamber its removal may involve considerable work. When removal is necessary it should be done with an eye to the vacuum gauge just as when the coating was applied so that as each leak is opened up its position will be noted by a pressure rise. After thorough cleaning of the region, the leaks may be repaired by whatever means are suitable.

10·6 Isolation of Components

Assembly of a complicated vacuum system without encountering one or more leaks occurs only rarely. When the system is composed of a number of components, a good plan of procedure is as follows. Starting with a vacuum gauge the components are added to the pumping system one at a time, and the effect of each addition is noted on the gauge. An increase in pressure will nearly always accompany each new component. A certain amount of this must be expected as a result of the increased surface exposed to the vacuum and the normal attenuation along the path to the pump. A little experience will enable one to judge whether the pressure increase is normal or whether it is due to imperfect cleaning or an actual leak in the component.

Many times it is desirable to pretest each component on a good pumping system before it is added to the more complicated system. In some arrangements stopcocks or valves may be placed between the components, and in such cases it is possible to make periodic checks of each section of the system. If these valves are not available, however, it may be necessary to remove each component completely in order to ascertain its contribution to a leaky system.

10·7 Analysis of Gas

There are times when identification of the gas in a vacuum system yields information as to the type of leak or impurity which may be present. When the pressures are high enough to give a glow discharge, spectroscopic methods may be applied. They may be as simple as the use of a pocket spectroscope, or it may be desirable to arrange for use of a spectrograph to detect and identify traces of gas.

If pressures are much lower the use of a mass spectrograph has become important.[5,6] This device is arranged to accept unknown gas, which it ionizes. A beam of these ions is directed through a magnetic field which gives each ion a deflection, depending upon its mass. Extremely sensitive means are provided for measuring the ion current in each component. This sensitivity makes the instrument useful in leak detecting.

In application it may be adjusted for a gas such as helium and used as an indicator on the vacuum system, the helium being applied over the external surface until an indication at the mass spectrograph denotes the leak. In another method the chamber suspected of leaking is filled with helium and then closed off. A tube connected to the gas inlet of the mass spectrograph is then used to search the surface, and as it nears the leak the escaping helium will enter the mass spectrograph and be indicated.

The mass spectrograph need not be restricted in its use to such a specialized case. It may also be used to detect and analyze other gases which may be present in the vacuum system. Although extremely sensitive, this method of leak detection is rather complicated and costly and probably is justified only in very special applications.

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6. R. B. Jacobs and H. F. Zuhr, *J. Applied Phys.,* Vol. 18, p. 34, 1947.

Chapter 11

METAL VERSUS GLASS VACUUM SYSTEMS

11·1 General Discussion

In the discussion to date, stress has been placed on the use of glass in the assembly of vacuum systems. More and more recently the necessary components have become available with which to assemble vacuum equipment in metal. Whereas, heretofore, apparatus using a high vacuum had been confined for the most part to laboratory surroundings, recent trends have indicated that vacuum equipment must be made available for a wider range of application. Such instruments as electron microscopes, for instance, cannot be confined to restricted laboratory usage if their greatest value is to be realized. The use of metal is indicated in such applications both because of its ruggedness and because of the accuracy which must be built into the equipment.

The glass systems have one advantage which the laboratory worker finds difficult to relinquish. It is the ability to see what is going on as evidenced by the color of the discharge phenomena and by observation of the mercury or oil as it circulates through the diffusion pump. These items mean nothing, of course, to the user of a piece of evacuated equipment, who is interested only in the normal operation of that equipment. Thus a well-engineered metal system which is operating satisfactorily is not necessarily at a disadvantage because of being completely enclosed.

11·2 Availability of Components *

The rotary forepumps which were discussed earlier are of metal construction and, of course, are equally suitable for either

* For up-to-date information on materials, components, and equipment reference may be made to the following annual directories: *Electronic En-*

metal or glass vacuum systems since rubber tubing is commonly used to connect them with the diffusion pumps, which may be either glass or metal.

Early diffusion pumps were always made of glass. Metal was sometimes used in the jet structure to insure greater accuracy in the jet tolerances. A wide variety of all-metal diffusion pumps is now available. Pumping speeds of a few liters per second to many thousand liters per second are available in either air-cooled or water-cooled structures. In this application there appears to be no fundamental superiority of metal over glass or vice versa. However, probably because of greater ease of construction, the larger pumps (those much above 100 liters per second in capacity) are made of metal.

Some of the gauges are readily adapted for use with an all-metal system. Ionization gauges and thermocouple gauges are available in metal envelopes as well as in glass. Traps are as easy to design and build in metal as in glass. Baffles are used chiefly at the diffusion pump inlet and are essential for either metal or glass pumps when inlet diameters above about two inches are being used. They are best used as close to the high vacuum jet as possible in order to catch back diffusing vapors and allow them to condense and drop down into the pump. Consequently, they are usually designed for and available with the larger pumps whether glass or metal. The baffles plates or disks are usually metal in any case.

Glass tubing between various components of the system has always made it possible to position the components securely and then bend the tubing to suit. In the case of a metal system soft copper tubing may be used, the small sizes in particular will take considerable bending, and larger sizes may be bent by filling the tube first with Woods metal, making the bend, and then removing the Woods metal by melting. Repeated annealing of the copper tubing in an oxygen-free flame will greatly facilitate its bending.

gineering Directory, "Electronic Industries," Caldwell-Clements, Inc., 480 Lexington Avenue, New York, N. Y. *Directory of Electronic and Allied Products,* "Electronics," McGraw-Hill Publishing Co., 330 West 42nd Street, New York, N. Y.

Flexible metal tubing, which is obtainable commercially in a wide variety of sizes, furnishes a much more flexible means of connecting components than the smooth copper tubing. It might be thought that the many convolutions in such tubing would slow down the pumping speed. Practically there is very little difference. When it is used, however, considerable care must

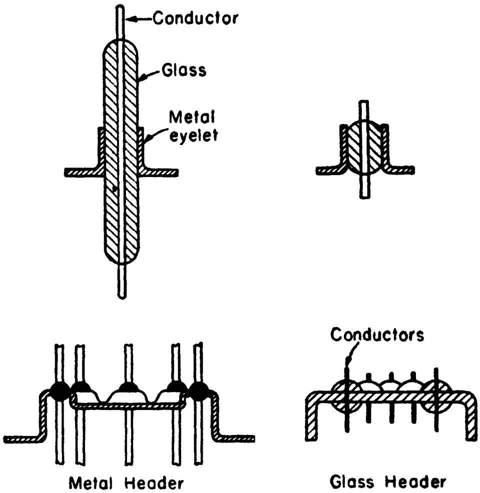


FIG. 11·1 Typical Metal-Glass Seals Commercially Available.

be exercised to insure complete removal of the lubricant used in its fabrication. Thorough cleaning with any good degreaser is quite adequate. Dilute nitric acid may then be used to remove the scale found on the inside of the tubing.

In the discussions of glass systems, methods were described for sealing wires through the glass walls for the purpose of conducting electricity to elements in the vacuum system. For this purpose, specially prepared wires are obtainable commercially or they may be made quite easily in the laboratory. In the metal systems glass-insulated conductors may be obtained so mounted in metal that the unit may be soldered or welded to the metal chamber. Many designs of these metal-glass seals are available in the forms of wire, eyelets, cylinders, etc. Multilead stems such as are used in radio tubes are commercially available

and are often quite useful in glass systems. The metal-glass "headers" used in metal tubes serve the same purpose for metal systems. These headers are available with a number of wires sealed in glass in a metal disk. The disk may be waxed, welded, or soldered to the vacuum chamber. Figure 11·1 shows some of the metal-glass seals available commercially.*

11·3 Materials

In preparing working chambers of glass, it is possible to obtain commercially many standard sizes and shapes of glass tubing and envelopes.

The electronics industry demands a steady production of a wide variety of bulb and tube shapes in sizes varying from miniature radio tubes to large cathode ray tubes and power tubes having dimensions of the order of several feet. The chemical industry also requires many standard glass shapes, large and small, in both Pyrex and soft glass. It is quite often possible to adapt an experiment to one of these standard shapes and eliminate a great deal of heavy-duty glass blowing.

In metal systems it is usually necessary to build up the shape desired. This often may be done by using standard commercial drawn tubing which is available in a number of cross-sectional shapes having dimensions of 6 or 8 in. or more. Suitable flanges, coverings, and openings must be provided. If the desired configuration is not available it must be built up from material available in other shapes, such as flat stock, or it must be cast.

Many difficulties may be encountered with either method. Castings may contain pinholes, voids, and other imperfections such as a general porosity which may make it impossible to obtain a vacuum. Sometimes it is possible to coat the surface of the casting in such a way as to seal these imperfections. For instance, dipping the part in molten tin is very satisfactory, or a coating of suitable varnish may be used.

When a chamber is fabricated from flat stock as, for instance,

* Electronic Sales Dept. Technical Products Division, Corning Glass Works, Corning, N. Y. Stupakoff Ceramic and Manufacturing Co., Latrobe, Pa.

rolled brass, two things must be kept in mind. First, the design must be such that the structure will maintain its shape under repeated applications of vacuum. Second, it must be a vacuum-tight assembly. As a rule, a flat-walled assembly which is merely soft-soldered is undependable unless the wall is quite thick or is re-enforced. The flexing and breathing of the walls due to the application of the vacuum will work the solder joints so that leaks will ultimately develop.

The use of hard solder, brazing, welding, or assembly with screws is usually essential and sufficient wall thickness should be provided. However, joints made by most of these methods are usually very difficult to make vacuum-tight. In a good method to follow the assembly is first made structurally satisfactory and then all the joints are sealed with soft solder or the entire structure is tin-dipped to provide sealing.

Some of the worst troubles attending the fabrication of a metal vacuum chamber come with the use of flux. All excess flux should be cleaned from the work thoroughly or corrosion may give rise to leakage and metal failure later. Many of the common fluxes are water soluble and may be removed by immersion in boiling water a short while. Other fluxes may require special treatment for removal. Since most commercial fluxes are trade secrets, the manufacturer should be consulted if there is doubt as to the proper cleaning technique.

Great care must be taken to avoid flux pockets. Such a pocket may plug so completely with flux that it will give a good vacuum reading after first assembly and test. Eventually, however, because of either its working loose or solvents used in cleaning or testing, such a flux pocket will become an intermittent leak and sooner or later will open up completely.

The materials most used for metal vacuum systems have been copper and brass because of their ready availability and ease of machining and soldering. Iron is quite satisfactory if its magnetic properties and susceptibility to corrosion can be tolerated. Stainless steel has been used with success, although in the past the difficulty in soldering it was a disadvantage. Aluminum and magnesium and their various alloys are being used more ex-

tensively, particularly as methods of welding and fabrication become better known.*

11·4 Assembly of Parts

Various methods of assembly of components using gaskets, both rubber and metal, and using various waxes were discussed earlier. These techniques are equally as applicable to metal systems as to glass. In general it is easier to obtain very high vacua in all-glass than in metal systems. This is because an all-glass system may be arranged to be given a more thorough bake-out than the usual metal system.

It is possible to get very good vacua with metal, however, as is evidenced by the success of metal radio tubes. Of course in this case much credit must be given the getter. In the ordinary metal system such as might be used with continuously pumped electronic equipment, it is usually difficult to pull down to much below 10^{-4} or 10^{-5} mm Hg. This may be due partially to the difficulty in outgassing exposed wax, grease, and rubber gaskets. Also thorough bake-out of combinations of metals having melting points varying upward from that of soft solder is not easy. Difficulty in locating all the extremely fine leaks which are nearly inescapable in a complicated assembled metal structure make attainment of a very high vacuum nearly impossible unless disproportionately large pumps are employed.

Happily for much electronic equipment, it is necessary to reach vacua only where the mean free path of the gas becomes large compared to the dimensions of the equipment. A vacuum of 10^{-4} or 10^{-5} mm Hg is satisfactory for use with electron microscopes, electron diffraction instruments, cathode ray apparatus, and similar high-voltage equipment. Of course this would not be at all satisfactory in a mass spectrograph, where the object of the instrument is to deal with traces of gas. Also the best possible vacuum is required in studies where surface conditions are critical. Experiments having to do with thermonic emission, photosensitivity, or secondary emission phenomena, for example, must be carried on under carefully controlled vacuum conditions.

* The Eutectic Welding Alloys Co., 40 Worth Street, New York 13, N. Y., specializes in low-temperature welding rods and fluxes applicable to nearly all the metals encountered in experimental electronics.

Chapter 12

SOURCES OF CHARGED PARTICLES

12·1 General Discussion

The latter part of the nineteenth century saw much experimental work carried on in the field of gaseous discharge. Starting with the particles encountered in that work, an amazing list of minute physical entities, some charged and some neutral, has been built up. The history and many of the theories concerned in this development are amply described elsewhere. Our aim at this time is to provide a working knowledge with which the experimenter may apply the charged particles. With this in mind we shall describe various methods of obtaining electrons and ions as well as the means by which they may be put to work.

12·2 Gas Discharge as Electron Source

A gas discharge may be initiated in a vacuum tube by providing a suitable potential to two spaced electrodes and sufficiently reducing the gas pressure below atmospheric. Pressures

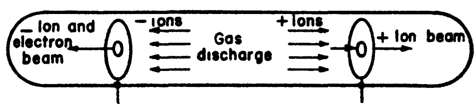


FIG. 12·1 Charged Particles in a Gas Discharge.

below 100 microns are commonly used. The discharge will be found to contain positive and negative particles. When the applied voltage is direct current, positive ions will proceed to the cathode while negative ions and electrons travel to the anode.

If either of the electrodes is perforated, some of the particles approaching that electrode will pass through it and emerge from the other side. In such a way, a source of that particular type of particle can be constructed (Fig. 12·1). If electrons are to be

generated and a narrow beam of electrons is desired, a single aperture in the anode is used.

If the cathode is made of a metal such as aluminum its bombardment by positive ions will result in the liberation of many secondary electrons with a minimum of disintegration due to the bombardment. These secondary electrons join the negative particles formed in the discharge and have velocities corresponding to the full discharge potential plus their initial velocities.

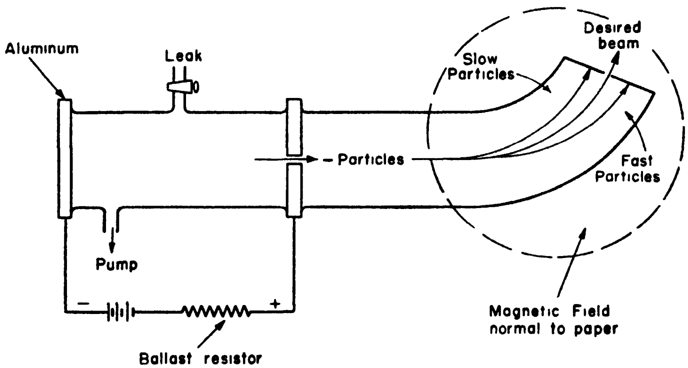


FIG. 12·2 Gas Discharge Source of Electrons and Negative Ions.

The discharge chamber is usually constructed so that it has its own pumping system and is connected to the main vacuum chamber only by means of the aperture through which the electrons issue. This constitutes a relatively slow leak, and a good vacuum may be maintained in the main chamber with suitable pumps. Since there will be some negative ions present the source will not be of pure electrons. This purification may be achieved when desired by separating the particles according to their masses by means of a magnetic field. Such a process will be described later.

The electrons issuing from the aperture in the anode will have velocities corresponding to the potentials acting upon them at their various points of origin in the discharge plus any initial velocities they may have had. Those coming from the cathode fall through the full discharge potential as mentioned above. Selection of all electrons having a narrow range of velocities is

possible by magnetic means just as in the separation of ions according to their masses. The selected electrons may be further accelerated or deflected as may be desired after entering the main vacuum chamber. Figure 12·2 shows a typical application of this type of source.

12·3 Cold Cathode Emission

When a sufficiently strong field is applied to a conductor, some of the loosely bound "conduction" electrons may be drawn forcibly from the conductor and accelerated along the field. Such a condition is called cold cathode emission, or autoelectronic emission, and occurs under fields of 10^6 volts per cm or more. Since this is an electrostatic field phenomenon we may find that conditions for its existence may be present even though comparatively low potentials are used. We know that field distribution conditions are markedly different for a potential applied between two planes as compared to one plane and one sharp point. Near a point the field becomes very highly concentrated. Thus a sufficiently sharp point may have associated with it extremely high fields even though the applied potential is relatively low.

At times a surface may contain microscopically small projections, and fields may be built up at these points to the extent of millions of volts per centimeter. Such conditions may exist on scratched surfaces and in the presence of particles of dust. Any condition wherein we encounter small radii of curvature is a suitable candidate for field emission of electrons.

In addition to the quality of the surface finish, field emission is dependent upon the type of material used as the cathode. The "breakdown" or "flashover" potentials of a few metals are shown in Table I. The effect of gas on field emission is generally to destroy the point from which the emission occurs. This results from the positive ion bombardment which is set up at the point by the high field concentration. Thus one of the difficulties encountered when it is desired to use the field emission from a point as an electron source is this deterioration of the point due to gas bombardment and disruptive field forces.

When the current is properly limited, the vacuum conditions are satisfactory, and a sufficiently stable point is available, this

TABLE I

HIGH-POTENTIAL BREAKDOWN RESISTANCE OF SOME METALS

	Voltage Breakdown Resistance in kv/cm	
	Polished surface	After breakdown
Al	200	163
Brass	230	110
Cr, Ni, steel	440	275

From E. Golz, *Jahrbuch der AEG—Forschung*, Julius Springer, Berlin, 1940.

type of emission may be used as a point source of electrons. Such a source when used with an anode of suitable geometry is capable of providing rectifying action. It also may be used in shadow microscopy (Fig. 12·3), where the object is placed in the electron beam as it leaves the point in a diverging cone.

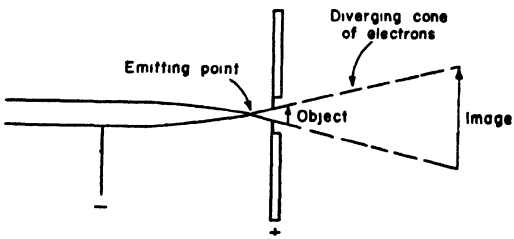


FIG. 12·3 Shadow Microscopy from a Point Discharge.

The true cold cathode effect is relatively independent of temperature and becomes increasingly so with higher fields. Some advantage may be had in the practical application of such a source, however, by operation at slightly elevated temperatures (Fig. 12·4). The main body of the pointed wire is heated by the passage of current, and the point is heated by conduction.

Tungsten is one of the more satisfactory metals for use as a point source. Molybdenum has also been used. The wire may

be sharpened to exceptionally small radii by several methods. It may be ground very satisfactorily if held with a rotating holder against a fine wheel. Very sharp points may also be obtained by plunging a red-hot tungsten wire into a stick of potassium hydroxide or sodium hydroxide. A number of passes into the caustic may be required to produce a satisfactory point.

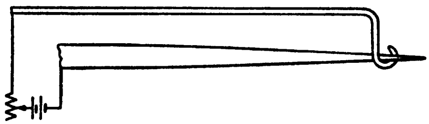


FIG. 12·4 Means of Heating Point for Increased Field Emission.

12·4 Hot Cathodes—Thermionic Emission of Electrons

The literature of thermionic emission is voluminous and to some extent controversial. No attempt will be made to treat the subject comprehensively. For such detailed reviews the reader is referred to Becker,[1] Dushman,[2] and Blewett.[3]

In the theories describing the emission of electrons from conductors it is assumed that an electron is freed from the conductor only after the expenditure upon it of a certain amount of energy. This energy, called the work function, customarily is denoted by ϕ and expressed in volts. It corresponds to the heat of evaporation and differs for different materials. It is extremely sensitive to surface conditions on the emitter. Table II shows the values of ϕ for some materials.

When a conductor is heated, as by the passage of current through it, its electrons find it increasingly easy to depart from the surface. In such instances a part or even all of the energy necessary to overcome the work function may be imparted to the electrons thermally. As a matter of fact, the electrons may even be ejected from the metal with a small excess energy of one volt or so. This gives to the electron what is known as initial velocity. Under such circumstances only the slightest collecting field is necessary to remove it from the vicinity.

Under certain conditions the collecting fields are limited and cannot remove the electrons as fast as they are emitted. The

excess electrons pile up in space outside the conductor, creating a region of negative space charge. This negative space charge tends to discourage further emission until it is limited to the value of current which the collecting fields can remove. Under these conditions the emitter is said to be "space-charge-limited" and practically no increase in current results from higher operating temperature.

TABLE II

EMISSION CONSTANTS FOR PURE METALS AND CARBON

Element	A	b_0	ϕ_0	I_T	T	ϕ_e
Carbon	5.9	4.57×10^4	3.93	2.8×10^{-3}	2,000	
Cesium	162.0	2.10×10^4	1.81	2.5×10^{-11}	500	1.91
Barium	60.0	2.45×10^4	2.11	1.4×10^{-3}	1,000	
Zirconium	330.0	4.79×10^4	4.12	8.5×10^{-5}	1,600	
Hafnium	14.5	4.10×10^4	3.53	2.8×10^{-4}	1,600	
Thorium	60.2	3.89×10^4	3.35	4.3×10^{-3}	1,600	
Columbium	37.2	4.66×10^4	4.01	1.16×10^{-2}	2,000	
Tantalum	55.0	4.86×10^4	4.19	3.20×10^{-3}	2,000	
Molybdenum	60.2	5.09×10^4	4.38	2.34×10^{-3}	2,000	
Tungsten	60.2	5.24×10^4	4.52	1.00×10^{-3}	2,000	4.54
Rhenium	200.0	5.92×10^4	5.1	3.4×10^{-5}	2,000	4.98
Iron	....	5.53×10^4	4.77			4.77
Nickel	1,380.0	5.84×10^4	5.03	5.1×10^{-7}	1,600	5.01
Rhodium	....	5.31×10^4	4.58			4.57
Palladium	60.0	5.79×10^4	4.99	3.0×10^{-8}	1,600	4.97
Osmium	....	5.45×10^4	4.7			
Platinum	17,000.0	7.25×10^4	6.27	9.2×10^{-10}	1,600	6.30

This table was kindly furnished by Dr. S. Dushman, Research Laboratory, General Electric Co.

When collecting fields are sufficiently high to sweep away instantly all electrons emitted, the emitter is said to be operating "temperature-limited" or "emission-limited." Under such conditions the nature of thermionic emission is such that greater and greater emission results as the operating temperature is raised.

The equation relating emission to temperature is commonly known as the Richardson-Dushman equation. One form is:

$$I = AT^2 e^{-b_0/T} \tag{12·1}$$

In this equation I = emission per unit area,
T = absolute temperature.

The constant b_0 is related to the work function of the emitter by the relation

$$\phi_0 = 8.618 \times 10^{-5} b_0 \text{ (volts)} \tag{12·2}$$

The constant A according to theory should be a universal constant. Actually this has been found not to hold in every case, there being some dependence upon the emitting surface. Thus to determine the temperature characteristic of an emitter both A and b_0 must first be determined. Table II gives these values for some materials. Column I_T gives calculated emission in amperes per square centimeter at T degrees Kelvin.

Equation 12·1 represents quite closely the relations existing under temperature-limited conditions. When the collecting field is incapable of removing the electrons as they are emitted and space charge limitation exists, the relation between the current collected and the applied potential is given by

$$i = KV^{3/2} \tag{12·3}$$

where K is a constant determined by the geometrical configuration. This relation assumes zero initial velocities.

The phenomenon of thermionic emission as described above appears to be fairly straightforward and will explain most experimental results. A few of the "effects" of secondary nature which are encountered in more detailed work will be mentioned here. For more complete information the reader is referred to the references given at the beginning of this section.

Schottky effect. Under temperature-limited conditions the emission is found not to be completely independent of the anode or collecting voltage. The effect is an apparent diminishing of the work function with increasing potential.

Electron temperature effect. The electrons issuing from a cathode by thermionic emission should have a Maxwellian distribution of velocities corresponding to the cathode temperature. Although the proper distribution appears to exist, the corresponding temperature has been found in some cases to

be 50 per cent or more higher than the actual measured temperature.

Shot effect. If the emission current is examined carefully it will be found to contain random high-frequency fluctuations. These fluctuations give rise to "noise" in circuit work.

Time effect. This effect mostly concerns oxide-coated cathodes which will be discussed later in the section. When such a cathode, well activated, is suddenly operated emission-limited the emission drops to a lower value. The decay characteristic is often of importance.

All metals to some extent have the property of thermionic emission, but not all are useful sources for electronic work. When the material is heated to the temperature at which satisfactory emission is achieved, it may be incapable of retaining its shape or dimensions for a satisfactory period of life. It may be that at satisfactory emission temperatures there will be excessive evaporation of the emitter or possibly it will melt down.

(*a*) *Tungsten.* Tungsten, molybdenum, and tantalum are among the most satisfactory thermionic emitters in the list of pure metals, tungsten being the more commonly used. Operating temperatures ordinarily are in excess of 2000°C.

Tungsten is a favorite emitter where its characteristics allow its application. Since it has no sensitive surface to be activated it may be used in apparatus which must be opened repeatedly to air. Such a filament is quite rugged and maintains its shape well throughout life even though evaporation may reduce its diameter to one-tenth the original. To its disadvantage is the high operating temperature with the resulting dissipation of large quantities of heat to the adjacent regions. In some applications the bright light is also undesirable. Tungsten becomes quite brittle after heating in vacuum and filaments may snap off at the slightest touch or even from the strains of expansion when heated and cooled.

Table III shows the operating characteristics of tungsten filaments of various sizes for a temperature of 2500°K (°C + 273°) and Table IV gives factors for converting this information to other temperatures. The data are given for filaments one centimeter in length. For these data to apply, the portion of fila-

ment under consideration should be sufficiently far from the terminal junctions so that end effects do not come into play.

TABLE III

CHARACTERISTICS OF A PURE TUNGSTEN FILAMENT ONE CENTIMETER LONG AT 2500° K

Diameter (inches)	Power Input			Emission		Life * (hours)
	A (amperes)	V (volts)	W (watts)	i (ma)	i/W	
0.0006	0.091	3.675	0.334	1.425	4.266	9.78
0.001	0.196	2.850	0.556	2.375	4.266	163
0.002	0.553	2.080	1.111	4.750	4.266	326
0.003	1.019	1.647	1.669	7.125	4.266	489
0.004	1.568	1.426	2.221	9.490	4.266	651
0.005	2.185	1.275	2.780	11.88	4.266	815
0.006	2.875	1.163	3.335	14.25	4.266	978
0.007	3.620	1.079	3.892	16.62	4.266	1141
0.008	4.42	1.015	4.45	19.00	4.266	1304
0.009	5.28	0.952	5.00	21.38	4.266	1468
0.010	6.18	0.901	5.56	23.75	4.266	1630
0.012	8.15	0.823	6.67	28.47	4.266	1955
0.014	10.25	0.762	7.78	33.21	4.266	2281
0.015	11.37	0.737	8.34	35.60	4.266	2442
0.018	14.95	0.672	10.00	42.71	4.266	2935
0.020	17.52	0.637	11.11	47.50	4.266	3260
0.025	24.48	0.570	13.89	59.35	4.266	4070
0.030	32.17	0.521	16.69	71.25	4.266	4890
0.035	40.54	0.482	19.45	83.02	4.266	5700
0.040	49.54	0.451	22.21	94.90	4.266	6510
0.050	69.22	0.403	27.80	118.8	4.266	8150

* Life is assumed here to be the hours required to evaporate 10 per cent of weight.

This table is calculated from data of H. A. Jones and I. Langmuir, *Gen. Elec. Rev.*, June 1927.

For short filaments the cooling at the ends is usually sufficient to make the tabulated data completely inapplicable to the filament as a whole.

Electron emission from tungsten is very sensitive to the presence of foreign materials on the surface of the metal. This is true even if the substance is present only to the extent of part of a single molecular layer. Some materials, including most gases, affect the emission adversely. Oxygen usually decreases the

TABLE IV

FACTORS FOR CONVERTING VALUES OF TABLE III TO CORRESPONDING VALUES AT HIGHER AND LOWER TEMPERATURES

Degrees Kelvin	Power Input			Emission		Life (hours)
	A (amperes)	V (volts)	W (watts)	i (ma)	i/W	
1000	0.160	0.054	8.63×10^{-3}	3.59×10^{-15}	4.16×10^{-13}	5.49×10^{24}
1200	0.238	0.100	2.38×10^{-2}	3.27×10^{-11}	1.37×10^{-9}	6.30×10^{17}
1400	0.330	0.166	5.48×10^{-2}	2.23×10^{-8}	4.07×10^{-7}	8.06×10^{12}
1600	0.434	0.256	0.111	3.11×10^{-6}	2.80×10^{-5}	1.63×10^{9}
1800	0.548	0.372	0.203	1.50×10^{-4}	7.39×10^{-4}	2.14×10^{6}
2000	0.670	0.513	0.343	3.37×10^{-3}	9.82×10^{-3}	1.16×10^{4}
2200	0.796	0.685	0.546	4.46×10^{-2}	8.15×10^{-2}	1.62×10^{2}
2400	0.932	0.886	0.825	0.389	0.471	4.64
2600	1.069	1.122	1.200	2.41	2.001	0.231
2800	1.210	1.390	1.682	11.90	7.070	1.81×10^{-2}
3000	1.356	1.690	2.295	47.5	20.7	2.09×10^{-3}
3200	1.505	2.030	3.060	160.5	53.5	3.04×10^{-4}
3400	1.660	2.410	4.000	477	119.2	5.68×10^{-5}
3600	1.814	2.830	5.150	1255	244.0	1.31×10^{-5}

The characteristics of various sizes of wires at 2500°K (Table III) are multiplied by the corresponding factors in Table IV to give the characteristics at higher and lower temperatures. This table is calculated from data of Jones and Langmuir, *Gen. Elec. Rev.*, June 1927.

electron emission from a pure tungsten surface. However, where other contaminants are already present the result may be an apparent increase.

In actual practice tungsten or other pure metals as emitters are customarily in the form of wire or ribbon. In some cases the electrons are drawn off radially from a long expanse of the wire, which may be arranged as single strands, coils, or long hairpins as shown in Fig. 12·5. When a "beam" of electrons is required, arrangements of coils or hairpins with properly shaped electrodes for acceleration are used. Examples are shown in Fig. 12·6. In all cases suitable allowance should be made for expansion of

the wire. Sometimes this may mean that extra electrode spacing is provided when measured cold. In long filaments, however, springs may have to be provided to take up the slack in the expanded wire.

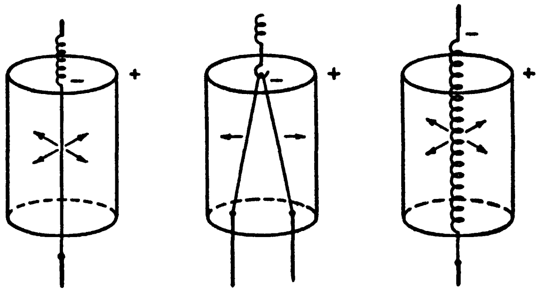


FIG. 12·5 Methods of Mounting Emitting Filaments (Emission Substantially Radial as Shown by Arrows).

(*b*) *Thoriated Tungsten.* The thoriated tungsten filament makes use of the sensitivity of tungsten to surface contaminants. Thoria (ThO_2), to the extent of 1 or 2 per cent, is included in the manufacture of thoriated tungsten. If the tungsten is heated to a sufficiently high temperature the thoria is reduced and thorium diffuses to the surface, where it evaporates. If the temperature is lowered the formation of new thorium will be slowed down and the diffusion and evaporation will assume different

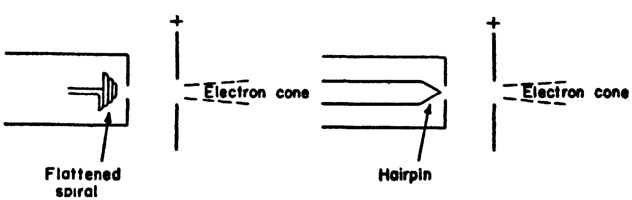


FIG. 12·6 Arrangements of Filaments to Produce Beam Source of Electrons.

rates. If the diffusion exceeds the evaporation a layer of thorium will begin to cover the surface.

If conditions are right, that is, a sufficient supply of reduced thorium is available and the right diffusion and evaporation rates

exist, an equilibrium condition can be maintained in which difuson just supplies the evaporated thorium and a monolayer of thorium covers or nearly covers the surface of the filament.[4] Under these conditions a fairly stable emitter is obtained giving approximately 2 amp per sq cm, roughly 1000 times the emission of tungsten at the same temperature.

Activation of a thoriated filament consists of flashing it for a minute or so at 2400°C or above to form a supply of thorium. The temperature should then be reduced to about 1750°C to 2000°C. In this temperature region the diffusion exceeds evaporation and the monolayer of thorium builds up. Actual operation of the filament is at about 1650°C, at which temperature both diffusion and evaporation rates are so low that the monolayer does not change appreciably with time.

Because of the difficulties attending measurements under these equilibrium conditions, consistent values of emission characteristics A, b_0, and ϕ_0 have not been obtained. For a normally operating cathode, ϕ_0 appears to be in the neighborhood of 3 volts. The results vary considerably with the fraction of the surface covered by the monolayer. The exact mechanism by which the thorium layer is able to enhance the emission of tungsten so greatly is unknown. Maximum emission has been found to occur when the monolayer covers about 70 per cent of the surface.

Satisfactory operation of a thoriated filament requires a good vacuum. The presence of gas may cause physical removal of the monatomic layer of thorium by ion bombardment or deactivation by chemical reaction of the thorium with oxygen or other active gases.

For satisfactory life from a thoriated filament the activation "flashing," that is, the initial heating in vacuum to 2400°C must be for such a time that a sufficient quantity of thorium is formed in the tungsten to supply that lost by evaporation during the filament life. Usually about a minute is a satisfactory flashing time. If, because of an insufficient reserve of thorium, the emission drops during operation the filament can be restored by reflashing and reactivation.

If a tungsten filament is flashed in a hydrocarbon atmosphere, a shell of tungsten carbide is formed over its surface as a result of the decomposition of the gas when it makes contact with the

hot tungsten. It has been found that the rate of evaporation of thorium from such a carbonized tungsten surface is much less than from pure tungsten. This means that for the same evaporation rate the filament may be operated at a higher temperature. Under these conditions the supply of thorium is enhanced and the filament becomes less susceptible to ion bombardment under poor vacuum conditions.

The structural techniques involved in adapting thoriated tungsten to a specific emission application are identical to those for pure tungsten since the material is available in the same form.

(c) *Oxide-Coated Cathode.* Nearly all radio tubes of the smaller sizes and a great many other industrial type vacuum tubes make use of the oxide coated cathode. In addition to this, it is a very suitable source of electrons in much laboratory work, yielding under ideal conditions as high as 200 ma per sq cm at operating temperatures of only about 900°C. The mixtures of materials used, the method of application, and the material of the base structure have many possible variations, and nearly all the possibilities have been investigated. As a result, there are a number of schools of thought on how to achieve best results.

Such cathodes usually consist of barium, strontium, and calcium oxides in the desired proportions applied to a metal support such as platinum or nickel arranged so that it can be heated. It is generally conceded that nearly equal amounts of barium and strontium give best results and only a small percentage of calcium is usually added. The materials may be mixed and applied as carbonates, nitrates, or other stable compounds and are reduced to the oxide during the processing of the cathode in the vacuum system. Barium is superior to both strontium or calcium in actual emission, but the addition of the strontium makes for a more stable cathode and improves the life.

The powdered chemicals may be mixed and applied to the cathode structure in a number of liquid carriers. One of the more common methods is to make a suspension in nitrocellulose lacquer. This suspension serves as a protective coating and may be stored for a considerable period of time without deterioration of the emitting materials. Assemblies containing cathodes coated with such lacquer suspensions may safely be washed as part of the final cleaning procedure. Water may also be used to sus-

pend the emitters, as may alcohol, amyl acetate, and ethyl acetate. These latter suspensions do not have the adhering qualities of the lacquer, and it is a little more difficult to get the coating to cling uniformly to the base metal.

Barium alone may be used as a cathode coating and may be applied as a carbonate or nitrate as described above or in the form of the azide. The azide (which should be handled as an explosive) may be applied in water solution. When the water evaporates, a layer of barium-azide remains which later may be heated and converted over to the oxide or used as a barium metal coating upon removal of the nitrogen of the azide. Another form for application of the emitter is the resinate. In this case the structure is heated in order to melt the resinate as it is brought into contact with it. The resulting coating may then be oxidized.

When a large number of oxide-coated cathodes are to be prepared it is nearly always desirable to set up methods by which uniform coatings of controllable thickness may be formed. Commercially, this is usually achieved by using the proper proportions of the compounds of barium, strontium, and calcium suspended in a lacquer solution. This coating is then applied by spraying or if the cathode structure permits, a dipping operation may be used. Thicknesses may be controlled to about one thousandth of an inch. If too thick a coating is applied, the surface will run cool under normal operating current or, conversely, excessive heater temperature will be required for normal emission. If the cathode is too thin, the cathode life will be short and emission may be spotty instead of uniform. Coatings of 3 to 5 thousandths of an inch thick give satisfactory results.

When only a few cathodes are to be prepared it may not be practical to set up elaborate methods, and very satisfactory cathodes may be obtained by hand-dipping or painting the material on the cathode. A small spray gun is quite useful if it may be operated with a sufficiently small amount of prepared materials. The drying time of the nitrocellulose lacquer may be controlled to some extent by varying the proportions of amyl acetate and ethyl acetate used as a solvent. Ethyl acetate will cause faster drying. The "fluffiness" or "compactness" of a

coating are somewhat dependent upon the drying rate, particularly in spraying.

The materials used as the basic structure for oxide-coated cathodes are generally platinum (with a few per cent of iridium, rhodium, or nickel), nickel, or some alloy. Many experiments have been made to investigate the effect on emission of the physical condition and chemical composition of the metal base carrying the cathode coating. Various chemical treatments, etching, polishing, painting, etc., have been tried. The addition of a small amount of titanium in the nickel base metal has been held to give improved emission. An alloy known as Konel (nickel, cobalt, iron, and titanium) is also claimed to have superior properties.

Regardless of the method of application of the coating, the procedure followed after the cathode is in the system and evacuated first converts the coating to the oxide. This is accomplished by raising the temperature of the cathode and is part of the activating procedure. The chemistry of this step depends upon the exact materials used, but in any case there is usually sufficient oxygen present to accomplish the oxidation. The heating also accomplishes the decomposition of the lacquer or other carrier. Considerable gas is given off in this process, and as a result flaking may occur unless the heating is done judiciously. Etching of the cathode base metal has been found to help if the flaking is persistent.

The condition of the cathode during its entire period of formation (activation) may be followed to a considerable extent by careful observation of the pressure in the system. Measurement of the emission is also helpful in this connection when that stage of the operations is reached. The fresh oxide coating as formed will be found to have not much, if any, emission. The methods of promoting that emission are varied. One school holds that the activation can be done by temperature variation alone. Another school favors holding the temperature constant and following an activation schedule by varied applied potentials. All combinations of these extremes are practiced, and actually it is found that different activation schedules, usually arrived at empirically, are set up for each different type of cathode structure and surrounding geometry.

A good general procedure to follow for most cathodes is as follows. When a good vacuum has been achieved the temperature of the cathode is brought up slowly and the pressure gauge is observed. The original evolution of gas may in some cases be sufficient to block action of the diffusion pump and the temperature should be advanced at such a rate that this does not occur.

This outgassing will be virtually completed at temperatures of about 900°C. At this point the temperature is advanced to 1100° or 1200°C for one-half minute to a minute and then reduced to between 900° and 1000°C.

It is assumed that all baking and outgassing of other tube parts has been completed at this point so that the cathode now to be activàted will not be poisoned later by gases from the parts.

An electron collecting field is then applied to the cathode by means of some adjacent electrode. If the electrode is very closely spaced to the cathode, a few volts will suffice. If it is at some distance, however, much higher potentials may be required. An ammeter of suitable rating should be in the circuit to measure the emission. As the potential is applied slowly, current will be caused to flow in the circuit. In some cases the initial velocity of the electrons being emitted may cause a slight negative reading on the instrument before the potential is applied.

As the potential is increased, the emission will be observed to increase steadily to some saturation value. When this value is reached, the potential should be backed down slightly and the cathode allowed to age for a few minutes. As it activates, the emission will gradually increase, and occasional checks will reveal that the saturation value has increased also. When the cathode has apparently stabilized, the potentials which will subsequently be used on the device should be applied. If the cathode temperature is reduced slowly the emission should at first remain constant, but should suddenly become dependent upon the cathode temperature (temperature limited) and begin to drop. The operating temperature (space charge limited) is just above this point and for normal cathodes should be roughly 850°C.

There are times when a well-coated cathode will refuse to give any emission under any procedure such as just described. Emission may be started in some such instances by allowing the gas pressure to build up to a few microns with the cathode heated and collector voltage applied. The gas may be furnished by bombarding some of the electrodes with high frequency or by raising the heater temperature to an abnormally high value for a short time. Care should be taken to avoid excessive bombardment which might remove the coating.

Many attempts have been made to establish satisfactory values of A, b_0, and ϕ_0 for oxide-coated cathodes. The results have not been in too good agreement because of the complex nature of such cathodes. The literature seems agreed that of the three oxides CaO, SrO, and BaO, the highest work function is that of CaO whereas BaO is the lowest of the three. Furthermore, such cathodes have work functions lower than those of either tungsten or thoriated tungsten. Values found for BaO vary from 1 to 2 volts, for SrO, from 1.5 to 2.5, and for CaO, from 2 to 3 volts. Mixtures of these oxides have values of ϕ_0 lying within these ranges.

Theories as to the exact mechanisms involved in the functioning of oxide-coated cathodes are as widespread as the techniques for applying and activating them. A few facts have been established. It seems certain that an activated cathode consists of a monatomic layer of active metal on the surface of the oxide. It also seems certain that as this layer of metal is lost by evaporation or other means, it is replaced from the oxide beneath it. No attempt will be made here to analyze the various theories of operation which have been presented.

An activated cathode is very sensitive to abnormal conditions. Excess temperatures or suboperating temperature may impair its life or efficiency. High voltages applied under temperature-limited conditions should be avoided. "Poisoning" of an activated surface may result if contaminating gases or vapors are present. In addition to "poisoning" the cathode by chemical reactions, their presence will insure ionic bombardment which will destroy the activated surface.

Oxide-coated cathodes may be used with good life in tubes having anode potentials up to approximately 10,000 volts. Oper-

ating potentials much in excess of that value usually cause shortened cathode life because of ion bombardment.

Usually it is not good practice to attempt to reuse a cathode which has previously been activated and then exposed to air. However, many times such cathodes may be reactivated to a satisfactory degree. If the emission requirements are not too great this may be done a number of times. The emission available usually decreases with each exposure, and at no time is it certain that such a cathode can be reactivated.

A number of methods are available for heating oxide-coated cathodes. The so-called direct method consists of applying the coating to a ribbon or a wire which may be heated by the passage of current through it. This case of actual operation of the coated wire as an emitter may make use of the design schemes described earlier in connection with tungsten wire or ribbon.

Commonly called the indirect method is the method in which the cathode structure is a cylinder, pillbox, or some other similar enclosed structure, the coating being applied to the desired outer surface. Heat is generated inside by means of an auxiliary coil of tungsten, molybdenum, platinum, etc. This may or may not be in electrical contact with the cathode but is usually arranged so that efficient heat transfer occurs.

Most radio tubes have cylindrical cathodes, the outer wall being coated with emitting material. Tungsten heaters suitably formed into spirals or long hairpins are provided with a baked coating of some refractory material such as magnesium oxide and are inserted in the cylinders. These indirectly heated cathodes have been called unipotential cathodes since they do not have the potential gradient along them which accompanies a directly heated cathode.

The use of heating currents of the order of amperes means that magnetic fields will be present and in some cases this may be undesirable. Reverse coil heaters are then used. In such heaters the wire is wound back parallel to itself, thus effectively canceling out the magnetic field. Figure 12·7 shows some heater and cathode designs. In some, emission is radially from the cylinder; in others "point" emission from the end cap is used to obtain an electron beam.

Although the above-mentioned methods of heating are more common, other possibilities may be used. For instance, a small coated disk may be satisfactorily heated from some distance by focused radiation from a heat lamp. If a suitable structure is

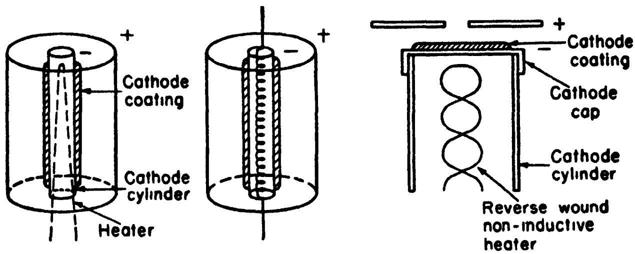


FIG. 12·7 Types of Indirectly Heated Cathodes.

arranged a cathode may be satisfctorily heated by electronic or ionic bombardment. These schemes obviously are used only when they are peculiarly suited to the experiment and for some reason the simpler methods are not applicable.

12·5 Photoelectric Emission

The discrete particles of light (photons) possess energy proportional to their radiation frequencies. A photon falling upon a surface can dislodge an electron from that surface if it can transmit to that electron sufficient energy to enable it to break through the surface forces, that is, to overcome the work function. This situation is described by Einstein's photoelectric equation.

$$h\nu = \phi_0 e + \tfrac{1}{2}mv^2 \qquad (12\cdot4)$$

where h is Planck's constant approximately 6.6×10^{-27} erg sec,
ν is the radiation frequency,
ϕ_0 is the work function (volts) (same as in thermionic emission),
e is the electron charge (e.s.u.),
m is the electron mass (grams),
v is the electron velocity (cm per sec).

Energy in excess of that necessary to release the electron gives it residual velocity and energy denoted by the second part of the

equation. If the photon energy is just equal to the work function, that is,

$$h\nu_0 = \phi_0 e \tag{12·5}$$

then ν_0 is referred to as the threshold frequency. Substitution of constants in equation 12·5 gives

$$\phi_0 = \frac{12{,}336}{\lambda_0} \tag{12·6}$$

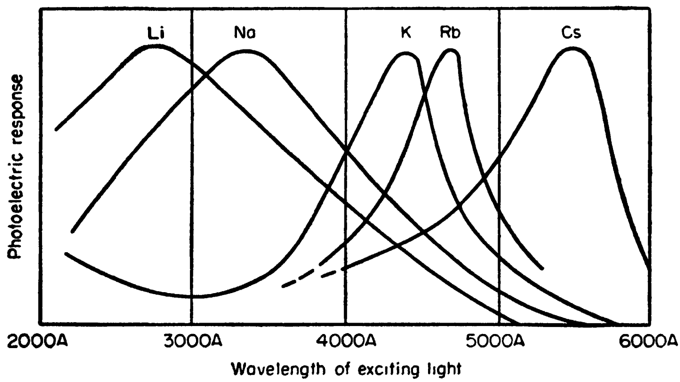


FIG. 12·8 Spectral Sensitivity Curves for Alkali Metals. (Hughes and DuBridge, *Photoelectric Phenomena,* p. 161, McGraw-Hill, 1932.)

where λ_0 is the wavelength in Angstroms (long wave limit) corresponding to the threshold frequency. Thus dislodging a photoelectron from a surface having a work function of 3 volts requires radiation of the wavelength 4112A (blue light) or less. Values of the work function found by photoelectric means are shown in column ϕ_e of Table II for a number of materials.

The efficiencies of radiation of different wavelengths to produce photoelectrons varies considerably. A curve showing this relation is known as a spectral sensitivity curve. Figure 12·8 shows the relative locations of such curves for the alkali metals. Because sufficiently reproducible and accurate data for comparing the efficiencies of the metals have not been obtained all the curves have been adjusted arbitrarily to the same height.

Similar curves for some composite surfaces are shown in Fig. 12·9. Here the relative efficiencies of the surfaces at various wavelengths are indicated by the heights of the curves.

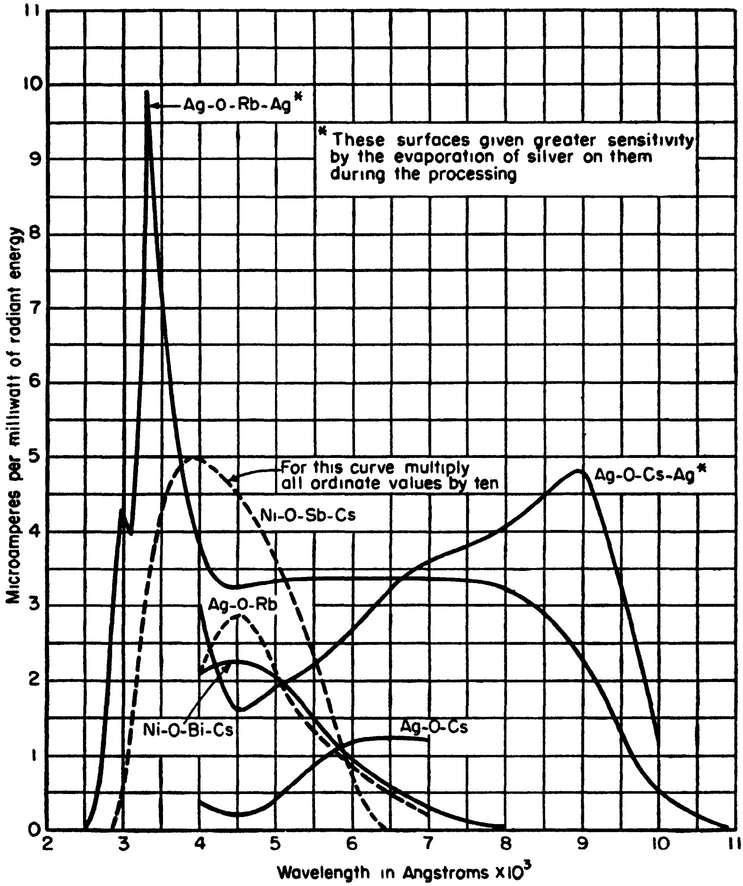


Fig. 12·9 Photoelectric Spectral Sensitivity Curves of Some Composite Surfaces. (Data for these curves kindly furnished by H. M. Smith, General Electric Company, Research Laboratory.)

If wavelengths greater than the long wave limit are used no photoelectrons will be produced. If shorter wavelengths are used to irradiate the surface, the electrons will have excess energies as given by equation 12·4. If these excess energies become quite

great as in irradiation by X-rays or gamma rays (corresponding to many thousands of volts) the energy expression $\frac{1}{2}mv^2$ must be replaced by that containing the relativity correction. Also the penetration of such radiation is such that more tightly bound electrons may be released and ϕ_0 increases by an amount equal to the energy required to remove the electron from the deeper energy level. Equation 12·4 is then replaced by

$$h\nu = w + m_0c^2\left(\frac{1}{\sqrt{1-\beta^2}} - 1\right) \tag{12·7}$$

where w is the new work function,
m_0 is the rest mass of the electron,
c is the velocity of light,
β is the ratio of the electron velocity to the velocity of light.

Photoelectric emission from gas is possible under these higher energy radiations.

Although nearly all materials are photosensitive to a degree, the alkali metals have been of most practical importance since they are sensitive to visible light (Fig. 12·8). The preparation of photoelectric surfaces from such metals is an art. The metals are characterized by low boiling points, and the methods of "distilling" described in Chapter 9 are commonly used. Large production may utilize the method of flashing prepared pellets such as is used in "gettering" tubes.

Such pellets for the introduction of cesium may be made by combining two parts by weight of cesium chromate with eight parts of zirconium and one part of aluminum. All the materials should be powdered and should be mixed as a paste in a suitable organic binder. To prevent ignition the components should be wet with the binder before mixing.

The active material must not be introduced until the base metal is properly cleaned and gas-free. Sensitization of the surface entails depositing the sensitive material upon it in the correct amount, either in the pure form or in some combination with the oxide or even other metals. Care must be taken to insure that excess material not be deposited on other parts which might adversely affect operation.

Often complex procedures involving baking, aging, and gas discharge treatments are necessary to prepare the type of surface desired. The progress of sensitization is frequently followed by actually making observations of the photoelectric response.

When once formed, a photoelectric surface is essentially independent of temperature so long as the surface is not actually changed. Although the effect of active gases is to decrease the sensitivity, the presence of inert gas may actually be used to increase the response of a photocell by five- or tenfold. A number of effects such as fatigue, time delay, and dark current will not be discussed here. For a more complete discussion of the subject, the reader is referred to other literature.[4, 5, 6]

Aside from the many ordinary photocell applications, the use of photoelectricity as an electron source is of specific rather than widespread interest. The thermionic sources described previously have led to designs in which satisfactory emission is obtained from relatively small cathode areas. The photoelectric current from a vacuum type cell varies linearly with the intensity of the light falling upon its surface. The efficiencies of a number of sensitive surfaces are shown in Table V. These are values which have been measured with such surfaces. It can be seen from the ranges of efficiencies given for each surface that wide variations in sensitivity are commonly encountered in attempts to duplicate photosensitive surfaces.

TABLE V

EFFICIENCIES OF SOME PHOTOSENSITIVE SURFACES

Surface	*Microamperes per Lumen* *
Ag-O-Cs	5–25
Ag-O-Cs resilvered	25–80
Ag-O-Rb	1–10
Ag-O-Rb resilvered	25–40
Sb-Cs	50–120
Bi-Cs	1–10

* Light source—incandescent tungsten lamp at 2870°K.

These data were kindly furnished by H. M. Smith, Research Laboratory, General Electric Co.

It can be seen from the table that photoelectric surfaces in combination with customary light sources are relatively inefficient. However, rather large surfaces of uniform sensitivity can be made, and probably the most important electronic applications have been in the field of image tubes such as the electron telescope and television camera tubes.[5] In such tubes a light image focused upon the sensitive area is converted to an electronic image of emitted photoelectrons.

"Transmission" type photosensitive surfaces may be made upon glass or mica by first providing the surface with a conducting, semitransparent layer of suitably inactive material either by evaporation of a metal or by one of the other methods more recently developed.

Methods have been worked out for treating glass in such a way as to retain high transparency while achieving good surface conductance. Information as to the characteristics of such glass and its availability can be obtained from the laboratories of the larger glass companies.

12·6 Secondary Electron Emission

An electron or other elemental particle striking a surface may give rise to the emission from that surface of one or more electrons. This phenomenon is called secondary emission, and the released electrons are called secondary electrons. These secondary electrons are emitted in random directions and with all velocities up to that of the incident electron beam. Most of the secondaries, however, will be found to possess velocities of only a few volts.

The number emitted per primary electron varies with the character of the surface and the velocity and angle of incidence of the primary electron. As compared with the photoelectric yield of not more than one electron per photon, ratios of secondary to primary electrons of 10 to 12 are not uncommon for sensitized surfaces. The work function apparently does not play the same part in secondary emission as in photoelectric and thermionic emission. Curves of secondary to primary ratio versus the accelerating voltage of the primary beam are shown in Fig. 12·10. As in the case of photosensitive surfaces accu-

rately reproducible results are the exception. The effect of combining surface materials is shown in Fig. 12·11.

It will be noted that the typical curve has two regions for which the yield is less than unity. One of these is for low voltages and the other for high voltages. Such a characteristic, together with the fact that both insulators and conductors yield

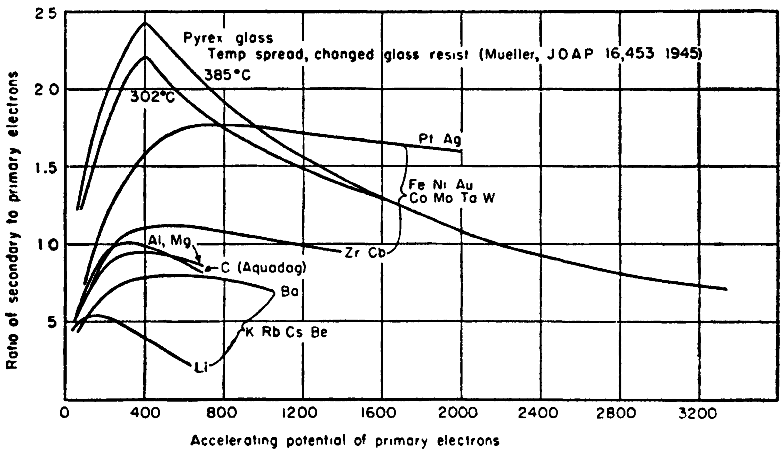


FIG. 12·10 Secondary Emission as a Function of Accelerating Potential of Primary Electrons. (Except where noted, data for these curves taken from H. Bruining, *Die Sekundär-Elektronen-Emission fester Korper.* Springer, Verlag, 1942.)

secondary emission, makes it important that the phenomenon not be overlooked in any electronic experiment. Regardless of whether it is purposely being put to work experimentally it nearly always is at work of its own accord and must be taken into consideration when electrons are moving about.

Thus if an insulator is bombarded by primaries capable of exacting a yield of greater than one and if a collecting field is at hand, that insulator will receive the bombarding beam in a stable manner. If the secondary yield is less than unity, however, the insulator will charge up and set up a repelling space charge which will let arrive at the insulator only as many primaries as are lost by secondary emission or other means such as leakage. The remainder of the beam will be deflected to some

other more receptive electrode. Such effects are usually erratic and are often encountered at the walls of glass bulbs and in the necks and at the screens of cathode ray tubes.

At times it is necessary to bombard an insulator with a beam which will not yield sufficient secondaries. Such conditions may

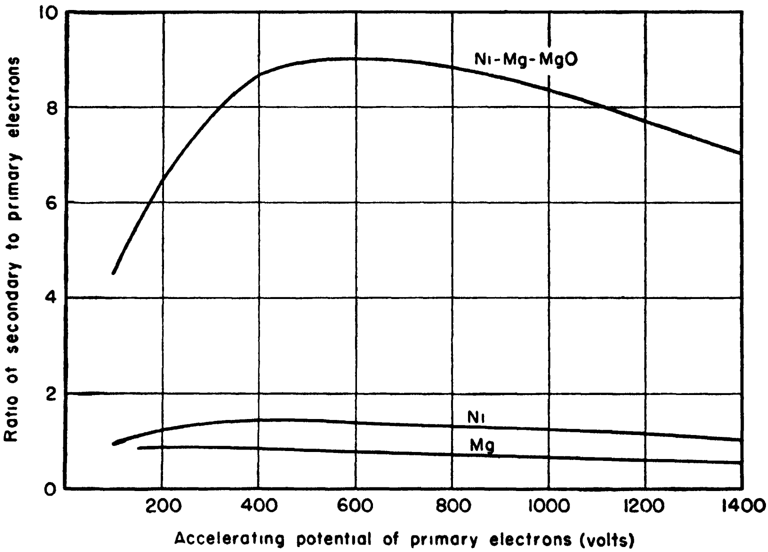


FIG. 12·11 Curve Showing Effect on Secondary Emitting Surface of Combining Materials. (Data for these curves kindly furnished by L. R. Koller, General Electric Company, Research Laboratory.)

occur during electron diffraction studies of insulators or at the fluorescent screens of high-voltage cathode ray tubes. The problem may be solved by simultaneously "spraying" the surface with electrons having velocities in the range which will give higher secondary ratios, thus removing the charge caused by the high-voltage beam.

These effects are not so troublesome in metal electrodes since circuit methods are usually available for removing the charge. However, a "floating" conductor can charge up and cause very peculiar results as is sometimes evidenced when a lead is broken or a connection neglected. In addition to the charging effects,

the secondaries leaving a bombarded surface may sometimes be collected with confusing results by a near-by electrode which just happens to have a suitable collecting potential.

Since these effects may be experienced under both very low and very high voltage electron bombardment and since the secondary ratio is very sensitive to surface contaminants, care should be taken to insure recognizing the presence of secondary emission. As shown by the curves of Fig. 12·10, proper choice of materials will help control it. It may also be minimized by proper control of the working beam and the elimination of all stray electrons.

Secondary emission as a practical source of electrons presents the basic consideration that one high energy electron can be made to generate a number of lower energy electrons. This attribute makes it attractive as a current amplifier. Multiplier tubes of many designs have been built for this application. They consist of an initial source of electrons, thermionic or photoelectric, with a number of secondary surfaces and a collector. The combination is arranged so that one electron leaving the cathode strikes the first surface and liberates n secondaries. These are accelerated toward a second surface where they give rise to n^2 electrons, and so on through a number of stages.

The remarks in the preceding section concerning the preparation of sensitive surfaces for photoelectric cells hold equally for secondary emitting surfaces. The procedures must be adjusted for maximum secondary emission, however, the requirements not being necessarily the same for the two types of emission.

12·7 Radioactive Emission

The charged particles emitted from radioactive materials are usually in such small concentration that they are not seriously considered as sources for electronic purposes. Because they are self-contained, however, there have been applications in which the charged particles of radioactive materials have been used to neutralize or minimize unwanted space charges on insulated surfaces and to aid in initiating gas discharges. An ionization gauge has been introduced commercially which contains a bit of radioactive material in place of a filament to provide alpha particles for ionization of the measured gas.

12·8 Ion Sources

Although we have seen that a number of electron sources have been developed to a rather high degree, the same cannot be said for ions. There are a number of phenomena which give rise to ions, but for the most part the efficiency is quite low.

(*a*) *Electronic Dissociation.* If gas molecules are subjected to bombardment by electrons, ionization by collision will occur. The efficiency of this process varies greatly with a number of parameters, including the kind of gas molecule, the gas pressure, the geometry of the container, and the velocity of the electrons. Both negative and positive ions may be formed. Figure 12·12 shows an arrangement sometimes followed. When this is the type of source, a stream of gas passes across the chamber and is removed by the pumping system. Electrons are directed into this chamber, causing ionization. The electric field is applied in such a way as to draw ions of the preferred charge to one side and out of the region. They are then collimated and employed as desired.

(*b*) *Ion Evaporation from Filament Surface.* It has been found that if cesium, potassium, or rubidium vapors or the vapors of their halides come into contact with a sufficiently heated (approximately 950°C) tungsten filament there will be re-evaporation of the metal as positive ions. The rule governing this is that the ionization potential of the metal should be less than the work function of the filament.[7]

(*c*) *Molecular Ovens.* The two preceding paragraphs described methods of ionizing a gas or vapor either by electron bombardment or by allowing the molecules to come into contact with a hot filament. In some cases the material is not in the gaseous form and is not vaporized at temperatures as low as the ambient operating temperature of the equipment. A molecular oven may prove a convenient means of introducing the molecules where desired. The theory of gas effusion and practical methods of utilizing it are available.[8]

The customary essentials are a capsule-like crucible with one rather small opening and a heater. The crucible is formed of metal or a refractory material, depending upon the nature of the material to be evaporated. The requirements are similar to those

met with in the evaporation of metals. The crucible must not react with the evaporated material and it must stand the temperature. Heating is usually accomplished with a heater wire coiled around the oven, but the other methods described under evaporation may be applicable. Figure 12·13 shows such an application.

After being charged, the crucible with its heater is arranged so that the beam of molecules issuing from the aperture in the crucible will pass into position for ionization. In some instances

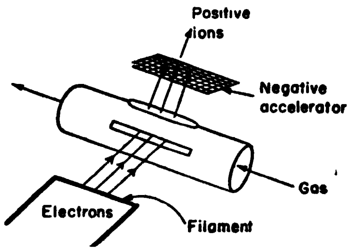


FIG. 12·12 The Generation of Ions by Electron Bombardment.

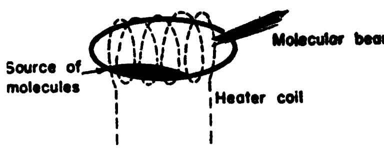


FIG. 12·13 Molecular Oven.

a considerable portion of the molecules will be found to be ionized as they emerge, possibly by some such mechanism as described in connection with the hot filaments, or possibly by thermionic ionization.

Control of the crucible temperature in turn controls the vapor pressure within it and accordingly the intensity of the molecular beam. To insure purity of the molecular beam suitable precautions must be taken in the preparation and cleaning of the parts and the material used to charge the crucible. The materials which may be vaporized in this manner are many. The method is quite commonly used in connection with such materials as the alkali metals and their salts.

(*d*) *Thermionic Emission of Ions.* It has become customary to assume that all negative particles leaving cathodes such as those previously described are electrons. This is far from the truth, however, and although the number of negative ions emitted from these sources is very small compared to the electron

emission, usable currents may be obtained by this method by magnetically separating the ion current from the electron current. Oxide-coated cathodes, for instance, will emit ionized oxygen copiously in both the atomic and molecular states as well as other gases. A large number of different negative ions have been reported [9, 10] emitted from hot cathodes of various types.

In addition to the negative ions accompanying thermionic electron emission there have been some indications that positive ions are similarly emitted. Barium and strontium ions have been reported. The effect is small, however. Thermal emission of positive ions from the metals chromium, molybdenum, tungsten, and tantalum has been reported.[11, 12]

(*e*) *Bombardment of Surfaces.* If conducting surfaces are bombarded by electrons or other ions, ions of either sign as well as electrons may be emitted. As a rule, the currents achieved in this way are too small to be of practical use, but the phenomena should be remembered as at times it may help explain investigational results.

(*f*) *Gas Discharges.* We have seen that both positive and negative ions, as well as electrons, are present in the gas discharge, and we have seen how the electrons may be utilized by providing the anode with apertures through which they may travel to arrive at the place of utilization. Similarly it is possible to make use of either the negative or positive ions. The positive ions will be normally attracted to the cathode and, if given the opportunity, will traverse a suitably placed aperture and emerge on the other side ready for application. The negative ions, in the presence of electrostatic fields, will follow the same paths as the electrons and appear outside the main gas discharge, back of an apertured anode.

(*g*) *Separation of Velocities of Masses.* The purity of a beam of charged particles obtained from a gas discharge is generally not very high as regards mass or velocity. A number of gases may be present in the discharge, and the ion-forming processes may give rise to multipli-ionized particles in the case of both positive and negative ions.

Since the paths of all particles of identical charge are the same in electrostatic fields it is impossible to separate particles of different mass by this means. However, particles of the same

velocity but different charge may be separated by electrostatic deflection, as may those having the same charge but different velocity.

Magnetic fields may be used to achieve mass or velocity separation. In such a case the heterogeneous beam of charged

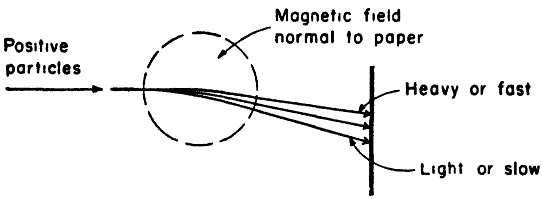


FIG. 12·14 Magnetic Separation of Particles.

particles is caused to traverse the field in such a way as to be deflected. This deflection will vary with the mass and with the velocity, the lighter and slower particles being deflected most. By proper choice of magnetic field, accelerating potential, and the geometry of the apparatus, particles either of the desired mass or velocity may be segregated for further utilization (Fig. 12·14). This process is utilized in many applications including the mass-spectrograph type leak detector described in Section 10·7.

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Chapter 13

CONTROL OF CHARGED PARTICLES

13·1 Mechanisms of Control

The activities of charged particles are directed and controlled fundamentally by magnetic and electric fields. These fields may be applied simply and directly or in complex combinations and configurations. The behavior of a charged particle in a given field configuration, either electric or magnetic or both, can be understood if the behavior of that particle in the simple field is known.

When a charged particle finds itself in an electric field it experiences a force tending to move it in a direction parallel to the electrostatic lines of force. The force tending to cause this motion acts upon either stationary or moving charged particles. Negative particles are drawn to the positive electrodes whereas the negative electrode attracts the positive particles. A charged particle moving parallel to electrostatic lines of force will suffer no deflection. If it is not moving parallel to the lines, however, the deflecting force will be that component of the field force which is normal to the direction of travel of the particle. In either case the velocity of the particle will undergo a change, either positive or negative, due to its acceleration by the field.

In the presence of magnetic fields charged particles exhibit slightly more complicated behavior. A magnetic field does not exert a force on a stationary charged particle. If the particle is moving parallel to the magnetic lines of force, it will experience neither deflection nor acceleration. A magnetic field applied normal to the direction of motion of a charged particle will exert a force upon it such as to produce motion at right angles to both the direction of the field and the direction of motion of the particle.

A good means of remembering the relation of the forces and motions in combinations of magnetic fields, currents, and beams of charged particles is as follows. It is customary to assume that when *electric current* flows in a wire in a direction away from an observer the lines of force surrounding the wire are set up in a clockwise direction, that is, in the direction of rotation

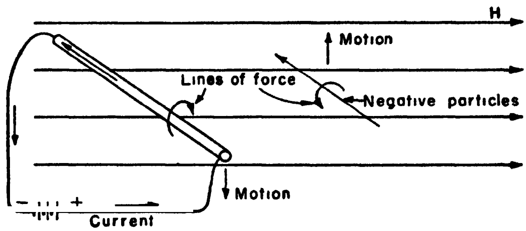


FIG. 13·1 Motion Resulting from Magnetic Deflection.

of an ordinary right-hand threaded screw advancing in the direction of the current. For *negatively charged particles* traveling away from the observer the action is negative to that of *current* flow; thus the lines are set up counterclockwise around a beam of such particles.

The deflection of such a beam of particles will always be such as to relieve the imaginary crowded condition of force lines caused by the lines of the beam adding to the lines of the main field. Motion will be in the direction in which the field is weakest owing to the main field being opposite to and thus canceling out the force lines of the beam. Figure 13·1 shows this relationship which can be applied to all such field reactions concerning beams of charged particles, current-carrying wires, and motors. The same fundamental reasoning is useful in connection with generator action.

A charged particle moving in a magnetic field at right angles to its direction of motion describes an arc. If there is a component of motion of the particle parallel to the magnetic field the particle will travel in a helical path.

The fundamental means of control described above are generally applied directly by means of electrodes, coils, and magnets of one form or another. However, the phenomena may be ap-

plied indirectly and in many instances unknowingly. For instance, a beam of charged particles traveling through space sets up magnetic fields just as though the current were flowing in a wire. If the beam current is sufficiently large this field may become sufficiently large to affect the experiment. Similarly space charge phenomena encountered where "clouds" of charged particles collect are actually electrostatic field phenomena since charged particles exhibit either attraction or repulsion for other charged particles by virtue of the electrostatic fields involved.

13·2 Accelerators

Charged particles are seldom found at rest in an electronic device. The mechanisms by which they are formed nearly always insure their having some residual velocity. The excess energy in a photon over that required to cause the emission of a photoelectron gives that photoelectron some velocity. Secondary electrons leaving the sensitive surface are found to have a wide range of velocities. If reflected primaries are included, the velocities will range from about zero up to that of the primary beam. In the case of a gas discharge source, the charged particles may have any velocity up to that given by the maximum potential used in the discharge. Thermionically emitted electrons may leave a cathode with velocities of the order of a volt or so as a result of energies imparted thermally. Likewise, the electrons emitted from radioactive materials have a wide range of velocities, depending upon the materials and the circumstances of the emission. None of these velocities is subject to much control. In some instances they may be disregarded as negligible with respect to the velocities to be imparted in the experiment. In other cases steps must be taken to filter out a suitable homogeneous beam of particles.

For all practical purposes, charged particles are accelerated as desired by suitable application of electrostatic fields. If beams of large cross-section area are to be accelerated, potentials are usually applied to plates; or if the main beam is to pass the accelerator, cylinders or screens of suitable mesh size are used. When small beams are being dealt with, the field may be applied in such a way that the particles pass through single small

apertures arranged in the plates to which the potentials are applied. Figure 13·2 shows how each of these methods might be used.

If apertures are small, care must be taken that they are in material of proper thickness. A small aperture in a thick material becomes in effect a long tube. It may be found to be difficult or impossible to direct a beam of electrons through such a tube. At certain current densities such tubes tend to choke off.

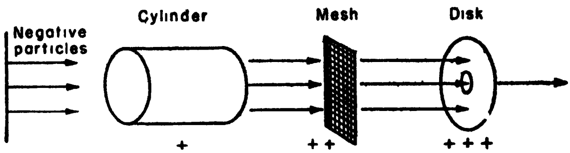


FIG. 13·2 Different Methods of Accelerating Charged Particles.

A good general rule is to use a material with thickness equal to or less than the diameter of the aperture which is to be used.

For all work involving charged particles at low velocities the energy relation is given by

$$\tfrac{1}{2}mv^2 = eV \tag{13·1}$$

where m, v, and e are the mass, velocity, and charge of the particle and V is the potential used to accelerate it.

By substitution of the proper constants this reduces to

$$v = 5.95 \times 10^7\sqrt{V} \tag{13·2}$$

where v is the velocity in centimeters per second and V is the accelerating potential in practical volts.

When the accelerating potentials are such as to give rise to velocities approaching the velocity of light, relativity corrections must be made and the energy relation becomes

$$\frac{\tfrac{1}{2}m_0v^2}{\sqrt{1 - \left(\frac{v}{c}\right)^2}} = eV \tag{13·3}$$

where m_0 is the rest mass of the particle and c is the velocity of light.

Figure 13·3 shows the relation between corrected and uncorrected velocities for a number of accelerating potentials. It can

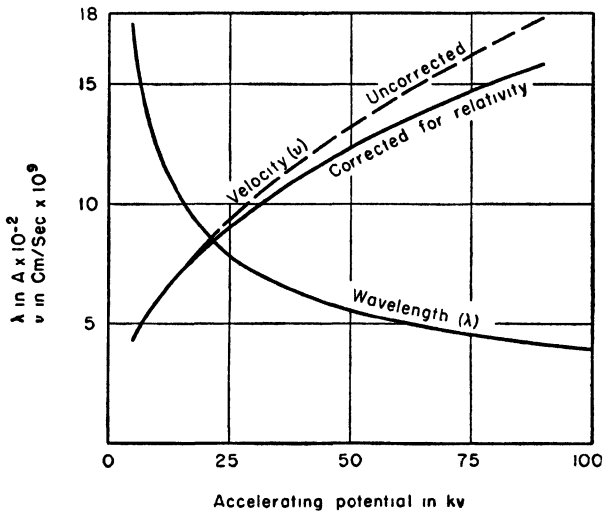


FIG. 13·3 Dependence of Electron Velocity and Wavelength upon Accelerating Potential.

be seen that at potentials not much above 50 kv the corrected velocities begin to differ appreciably from the uncorrected.

Figure 13·3 also shows the relation between the wavelengths of electrons and their accelerating potentials as obtained from the de Broglie relationship

$$\lambda = \frac{h}{mv} \tag{13·4}$$

where λ is the wavelength, h is Planck's constant, and m and v are the mass and velocity of the electron, respectively. In practical units this equation reduces to

$$\lambda = \frac{12.3}{\sqrt{V}} \tag{13·5}$$

where λ is the wavelength in Angstroms of the electron and V is its accelerating potential in practical volts. For potentials

above 50 kv it may be desirable to apply the relativity correction.

13·3 Electrostatic Lenses and Deflectors

The direct force which an electrostatic field exerts on a charged particle may be used for deflection purposes as well as acceleration. Of course, electrostatic deflection and acceleration are actually concomitant. In electronic terminology this fact is sometimes overlooked because the applications usually minimize one effect or the other. Depending upon the nature of deflection desired, electrodes must be devised to furnish as closely as possible the shape of field required at the proper points in space. Conductors in the shape of rods, rings, or plates may be suitably shaped to achieve this purpose.

The most common application of electrostatic deflection is that in which a beam of charged particles is passed through the field between a pair of parallel plates. Such deflection plates, for instance, are encountered in cathode ray tubes such as are used for television or oscillography. Such applications usually require deflection in two directions normal to each other so that either two sets of deflection plates or two magnetic deflecting fields or a combination are used. Figure 13·4 shows the arrangement using two sets of plates.

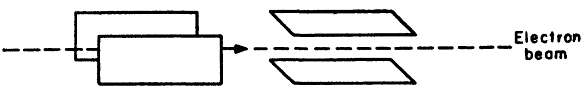


FIG. 13·4 Arrangement of Deflection Plates for Two-Dimensional Electrostatic Scanning.

The motion of a particle moving through such an electrostatic field is that of a projectile in a gravitational field and the deflection is dependent upon the mass, charge, and velocity of the particle as given by the equation

$$d = \frac{eEl^2}{2mv^2} \tag{13·6}$$

where the geometry is as shown in Fig. 13·5. The deflection h at a distance from the plates is given by

$$h = \frac{l(x - l)E}{2V_{acc}} + \frac{El^2}{5V_{acc}} \tag{13·7}$$

When x is large compared to l, this reduces to

$$h = \frac{xlV_s}{2SV_{acc}} \tag{13·8}$$

When deflections are small and the beam passes close to the center of the space between the plates there is little distortion. When the beam is passing through the plates in the region near their edges, however, distortion and defocusing effects are introduced due to the non-uniformity of the field in these regions.

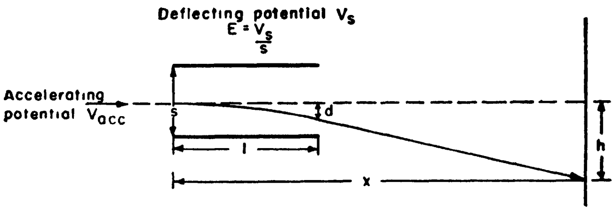


FIG. 13·5 Electrostatic Deflection.

In cathode ray tubes with two sets of deflection plates this condition is reached in the last pair of plates when the beam has been given maximum deflection by the first pair. Various schemes of minimizing this distortion include folding one of the plates at its ends, thus nearly closing the end gap (Fig. 13·6), and providing auxiliary guard plates with suitable potentials. The first pair of plates is quite often arranged in some non-parallel fashion to allow exit of a highly deflected beam.

The action on charged particles of the electrostatic deflection field with parallel plates may be likened to the action of a prism on a beam of light. Just as the refractive effect of a prism may be shaped in axially symmetrical fashion to give optical lenses, axially symmetrical arrangements of electrostatic fields behave in an analogous way to glass lenses. Beams of charged par-

ticles passing through such axially symmetrical fields are subject to convergence, divergence, and image formation and also the various aberrations encountered in "glass" or "light" optics.

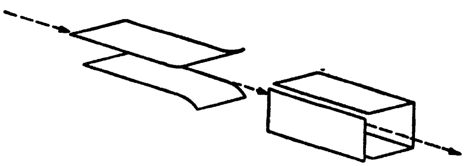


FIG. 13·6 Arrangement of Plates for Improved Deflection Qualities.

Field distributions satisfactory for providing lens action are commonly obtained using apertured disks and cylinders as electrodes. Figure 13·7 shows the paths taken by electrons through

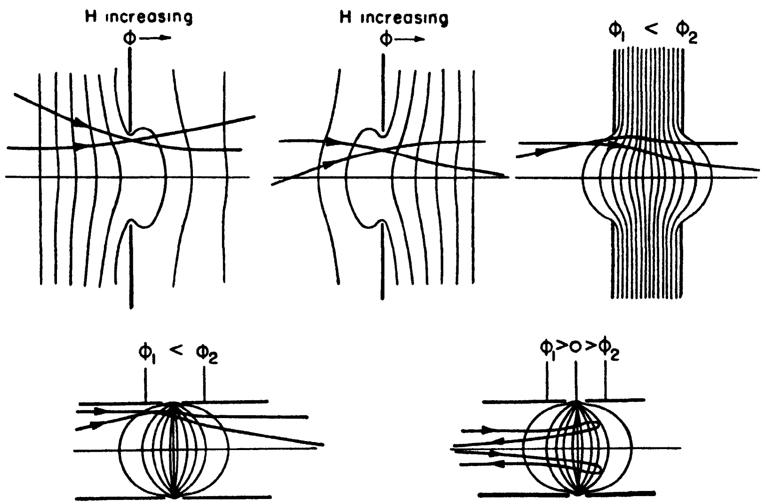


FIG. 13·7 Electron Paths in Several Types of Lenses. (From Ramberg and Morton, *J. Applied Phys.*, **10**, 472, July 1939.)

several lenses employing single or double electrodes. It is not essential that the diameters be identical in the case of either the apertures or the cylinders. The general lens action will remain the same although the lens power may change.

A particularly useful lens is the unipotential lens shown in Fig. 13·8*a*. It is operated with the center electrode either positive or negative with respect to the outer electrodes. This lens is nearly completely analogous to the double convex glass lens. Its focal length is a function of the electrode spacing, the diameter of the center aperture, the potential applied to the lens, and the velocity of the electrons. If the electron accelerating potential is the same as that applied to the lens the focal characteristics depend only upon the electrode geometry. Figure 13·9 shows typical curves of the relationship of focal length to geometry.

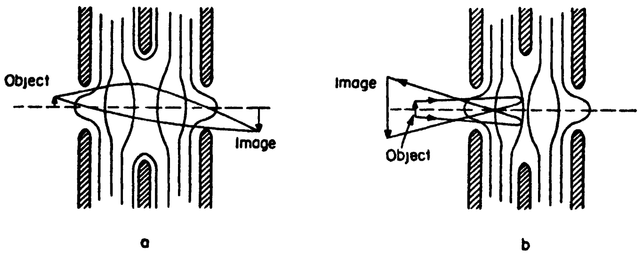


FIG. 13·8 Three-Electrode Unipotential Lens.

As customarily used, the lens must be classed as a thick lens, although simple thin lens formulae may be used to a fair approximation. If the center electrode is made sufficiently negative the electrons are turned back and the device becomes an electron mirror (Fig. 13·8*b*).

In devices such as the electron microscope, which require very short focal lengths, apertured disks may be preferable to cylinders in order to make possible the placing of the object or specimen in the most efficient position.

Aside from aberrations, which are a problem of electron optics rather than technique, electrostatic lenses have a number of advantages and disadvantages. The optical characteristics of such a lens are dependent upon its geometry, the lens potentials, and the electron velocity. If the lens potential and the accelerating potential are identical or vary in the same way the focal characteristic of the lens stays fixed regardless of voltage. The optical

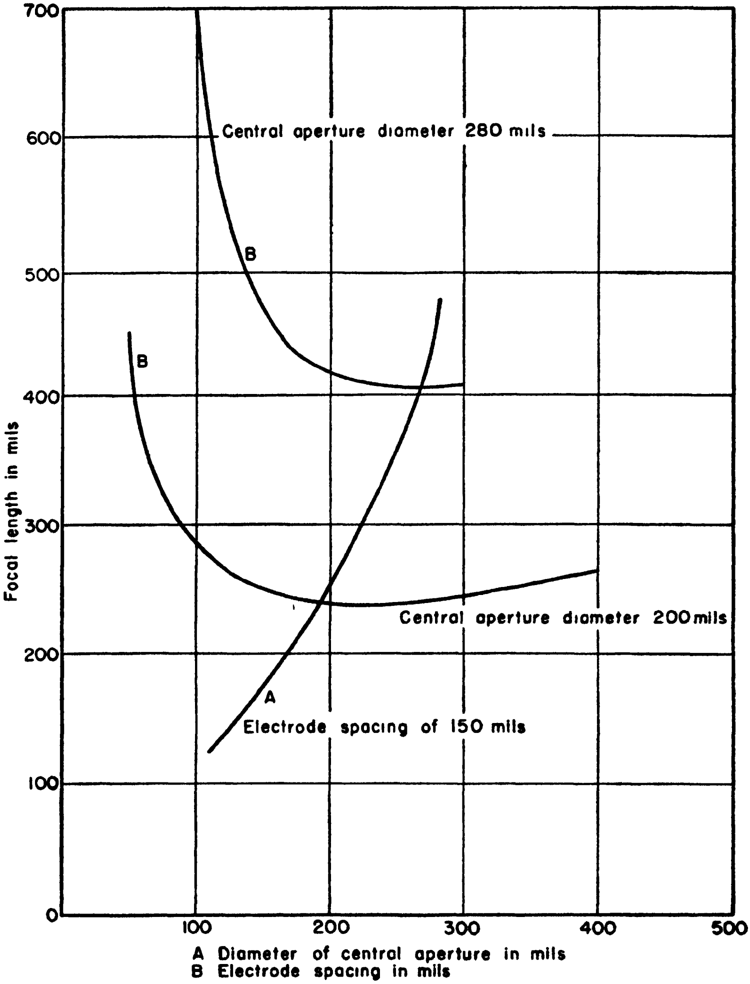


FIG. 13·9 Relationship of Focal Length and Geometrical Parameters in Unipotential Lens.

qualities of an electrostatic lens depend to a considerable extent upon the accuracy of construction and assembly. The care required to machine and assemble such a lens is offset by the fact that the lens stays fixed after construction.

When high voltages are used, problems of insulation, surface leakage, and flashover are encountered. When very low voltages are to be used, trouble may be encountered with deflections of the beam due to charging up of contamination on the edges of apertures.

For satisfactory lens operation apertures should be smooth and round with burrs removed. Cylinders should be straight and round and of wall thickness so that they hold their shape throughout assembly and processing. Surfaces should be smooth and edges clean. For high-voltage work the surfaces should be polished and all edges should have a good radius of curvature. Materials which may become magnetized should be avoided or at least they should be used with the full knowledge that they may become magnetized during the operation of the device, either by some external field or, in some instances, as a result of the magnetic field set up by the beam of charged particles.

13·4 Magnetic Lenses and Deflectors

Since magnetic fields exert no accelerating force on stationary charged particles their useful action becomes purely one of deflection of moving charged particles. For this, it is necessary that there be a component of the magnetic field normal to the direction of motion of the particle.

When a beam of charged particles enters a magnetic field at right angles to that field, it suffers a force tending to deflect it at right angles to both the field and its direction of travel. If the path of the particle in the field is comparatively short, it will travel in an arc path in the field and leave the field in a new direction.

The deflection of a negatively charged particle by a magnetic field is shown in Fig. 13·10. The equation governing this deflection is

$$y = \frac{mv}{eH} - \left(\frac{m^2v^2}{e^2H^2} - x^2\right)^{1/2}. \qquad (13\cdot9)$$

where the quantities are expressed in electromagnetic units. For electrons the deflection d at a distance l from the field, where x is small compared to l, reduces to

$$d = \frac{0.297Hlx}{V^{1/2}} \tag{13·10}$$

where H is in gauss and V is in practical volts. If the extent of the field is sufficient, as determined by the other parameters in

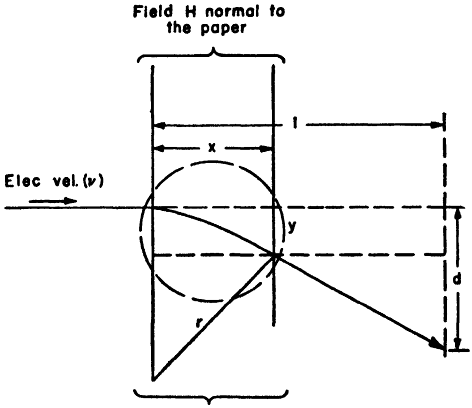


FIG. 13·10 Magnetic Deflection.

equation 13·9, the charged particle may not emerge but may continue around and around in a complete circle, the radius (in centimeters) of which is given by

$$r = \frac{mV^{1/2}}{eH} 5.94 \times 10^7 \tag{13·11}$$

where e and m are in electromagnetic units, V is in practical volts, and H is in gauss. The time required to complete each rotation is independent of the particle velocity and is given by

$$t = \frac{2\pi m}{He} \tag{13·12}$$

If a charged particle is moving in a uniform parallel field, in such a way that its direction of motion is parallel to the lines of

force of that field, no deflection will result. If the particle deviates in the slightest from its parallel path, the effect of the magnetic field on it is to force it into a helix, which will lead it back to a point intersecting the line of force which it left. This action will be continued through any number of helices, depending upon the length and strength of the magnetic field and the character of the charged particle. The result then is that, if

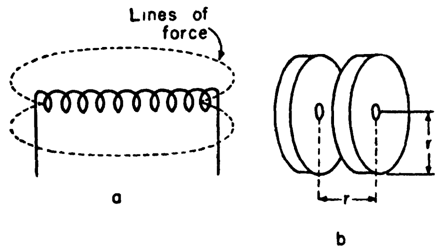


FIG. 13·11 Means for Producing Uniform Magnetic Fields.

the beam is substantially parallel to start with, small deviations will be restricted by the magnetic field in such a way that the beam remains parallel while in the field.

As just explained, each particle describing a helix passes again and again through the same line of force. The diameter of the helix is dependent upon the component of velocity normal to the lines of force (equation 13·11), whereas the axial distance traveled by the particle between each complete loop is proportional to the axial velocity component. Therefore, all particles leaving a point with the same axial velocity component will join at a corresponding point down the axis, regardless of the diameter of their various helices (that is, regardless of their radial velocity components).

Thus within a uniform field it is possible to have along an axial line any number of images, erect and of unity magnification.

Two convenient means of establishing uniform fields are by the use of the long solenoid and Helmholtz coils (Fig. 13·11). The solenoid is particularly valuable when it is desired to send

a beam of particles on a path parallel to the lines of a uniform magnetic field. The field at the center of such a coil is given by

$$H = 0.4\pi nI \tag{13·13}$$

where H is in gauss, n is the number of turns per centimeter, and I is the current in amperes. It must be remembered that a uniform field of this value exists only near the center of the coil, dropping to about one-half this value at the ends.

The Helmholtz coils are particularly useful when a beam of particles is to be sent transverse to the field. The Helmholtz arrangement consists of a pair of thin pancake type coils having an effective radius of r cm parallel to each other a distance r apart. The field at the center of the region half way between them is extremely uniform. The field strength in gauss is given by

$$H = \frac{0.286\pi I}{r} \tag{13·14}$$

where I is in amperes.

The design of deflection coils has been highly developed in the attempt to get distortionless deflection for television purposes. Some of the designs use iron cores and nearly all consist of four coils, opposite pairs being used to give deflections at right angles. Figure 13·12 shows the basic arrangement of coils. In actual practice there are special corrections for the distortions at maximum deflection and the coils are wound and assembled with an eye to compactness.

In Fig. 13·11 it is seen that the lines of force, although parallel in the interior of the solenoid, diverge symmetrically as they leave the ends. The reaction of the parallel force lines on diverging electrons producing a lens action of unity magnification was described. Conversely, if axially parallel electron beams pass through the non-symmetrical end fields of such a solenoid, a lens action is experienced. In fact magnetic lenses consist of solenoids shortened to the extent that the end effects predominate. The shortening is still further accentuated by enclosing the coil in an iron case which has a gap around its interior through which the flux expands to form a focusing field (Fig. 13·13).

Such lenses have the ability to form electron images just as does the unipotential electrostatic lens. However, a certain amount of rotation of the image occurs in all but very thin magnetic lenses and those corrected for rotation by the use of two coils in series along the axis, but wound in reverse directions. This rotation means that the image formed may be erect, inverted, or at some intermediate position.

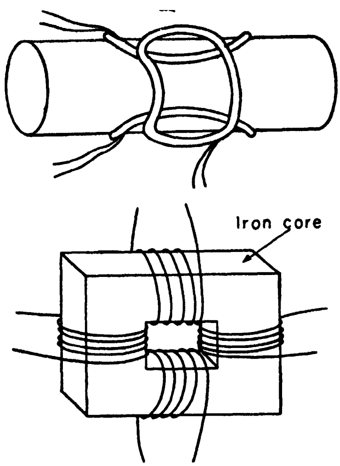


FIG. 13·12 Magnetic Coils for Two-Dimensional Scanning.

The characteristics of a magnetic lens are difficult to calculate as the mathematics becomes quite involved. The amount of image rotation is proportional to the ratio of field strength to electron velocity whereas the power of the lens is proportional to the square of this ratio.

Although an ordinary short solenoid can be used as a focus coil, it is possible to achieve a more symmetrical field as well as to reduce interference of the extended unused portion of the field by enclosing the coil in an iron case as shown in Fig. 13·13. Such coils may be used over a wide range of conditions. For instance, a useful coil is made of 4500 turns of No. 29 wire wound to a coil length of $1\frac{5}{16}$ in. and an internal diameter of 2 in. When this is enclosed in an iron case with about a $\frac{3}{8}$-in. gap, it can be used to focus to distances as short as a few inches, electron beams accelerated by voltages up to 50 kv or more.

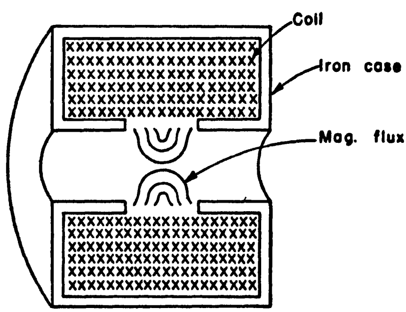


FIG. 13·13 Iron-Cased Focus Coil.

Where the field distribution is of greater importance it may be possible to place carefully machined iron poles in the vacuum system and energize them by surrounding coils placed outside the vacuum wall (Fig. 13·14). This scheme is common in electron microscope applications.

Permanent magnets make useful focusing devices. It is usually desirable to arrange the magnetic circuit in such a way that an easily made and energized magnet is combined with a prop-

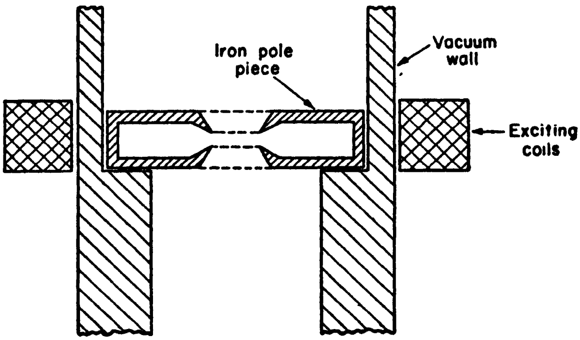


Fig. 13·14 Magnetic Lens Having Pole Piece Energized by External Magnet.

erly shaped magnetic circuit to give the required flux distribution. This reduces stray flux and concentrates the focusing flux where it is needed. Figure 13·15 shows typical arrangements. The elements are shown somewhat separated for clarity. Mechanical means for varying the strength of the field in the gap are possible.

There is a tendency for magnetic poles to decrease in strength and particularly to shift. Designs which encourage this shifting should be avoided. For instance, the arrangement shown in Fig. 13·15*a* is bad. The poles will shift in time until Fig. 13·15*b* results. Probably a cross-section similar to that in Fig. 13·15*c* is superior in this respect. Soft iron pole shoes should be used wherever possible as they tend to smooth out irregularities in the poles of the magnets.

In considering the type of lens to be used in an application, that is, electrostatic versus electromagnetic, the following factors

may be considered. The magnetic lens operates by means of applied current in a coil. The strength of such a lens does not bear the same relation to the potential source as in the case of an electrostatic lens. If variations occur in the supply it is not

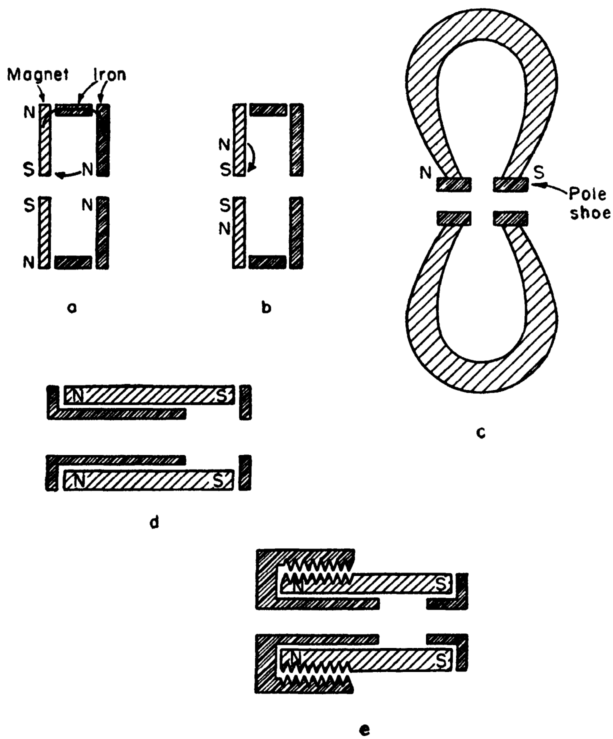


Fig. 13·15 Permanent Magnet Applications for Beam **Focusing.**

practical to arrange that corresponding variations in lens strength compensate for velocity variations of the charged particles as can be done with electrostatic lenses. The result will be that of a varying focal length, and the effect on a beam of charged particles will be a corresponding "fuzzing" of the control or focus. This effect may be obviated by stabilization of both the current of the lens and the accelerating potential of the charged particles. With permanent magnet lenses the ac-

celerating potential alone is required to be stabilized, of course.

An advantage of a magnetic lens is that it may be exterior to the vacuum system and thus may be adjusted mechanically for alignment purposes. On the other hand, this means that the magnetic focusing field is subject to extraneous magnetic effects. These effects may be minimized by proper use of shielding and by forming the lens field with carefully shaped pole pieces placed close to the beam.

None of the difficulties accompanying high-voltage operations is encountered with magnetic lenses since the high voltage does not appear on the lens directly. However, when small masking apertures are used the charging effects due to aperture contamination will be met with just as is the case with electrostatic lenses.

Electrostatic lenses are subject to easy control electrically as are magnetic lenses, although physical adjustment and line-up after assembly is usually not possible. In addition, lenses such as the unipotential type may be operated, if desired, with focal properties fixed regardless of electron velocity. Careful mounting and accurate machining are essential for a good electrostatic lens. High-voltage operation brings with it insulator problems which are not encountered with magnetic lenses. Electrostatic lenses are essentially self-contained, whereas the stray fields of magnetic lenses can be objectionable.

Where the conditions of the experiment do not dictate the choice of lens, it is quite probable that equally good results will be obtained with either.

In the case of either electrostatic or electromagnetic control of charged particles, the least controlling force is necessary when it is possible to place the electrodes close to the particles which are to be affected. Less voltage will be required to establish a given field with close-spaced plates than with plates of greater separation. Similarly, the power required to deflect the beam magnetically when the coils are close spaced is considerably less than that required for greater separation.

The effect of disturbing magnetic fields must be guarded against constantly in electron beam work. Magnetic sources within the experiment are difficult to shield and when possible

are best handled by removal as by choice of different materials. Shielding from outside sources is usually possible if the field is not too strong. The earth's field, which in some cases is sufficiently strong to interfere, can be easily shielded out whereas fields from devices such as variable voltage control transformers and voltage-regulating transformers are usually quite strong and may require their removal from the vicinity. When shielding is used it should be remembered that a number of spaced layers or shells of thin shielding material are far superior to the equivalent thickness of solid shielding metal. The spacing means may be any non-magnetic material.

13·5 Electron Guns

The term "electron gun" is rather loosely applied to an electron source together with whatever system of electrodes is required to shape an electron beam for use in the electronic application at hand. The term has attained its greatest prominence in connection with cathode ray tube structures, although it certainly is not restricted to that field.[1, 2, 3] Figure 13·16 shows a number of electron gun types that are commonly used. With the exception of Fig. 13·16*e* they are all axially symmetrical, consisting of cylinders and disks. The sketches show the electrode edges which would be exposed in a plane passing through the axis. The approximate ray paths are shown for electrons. The first electrode passed by the electrons is usually operated as a control grid at negative potential, although in some gun designs a slight positive potential is required on this electrode. The dimensions and voltages shown in the sketches are approximate only. If these values are used as a reference, the design can be adjusted for other operating potentials. Figure 13·16*e* is the same type as Fig. 13·16*d* except that it does not have the axial symmetry and a flat beam of electrons is focused to a line instead of the round spot of the other guns.

It is usually assumed that the fields in the cathode grid region of these guns give rise to an electron crossover point as shown. This may occur anywhere in the grid anode region and as a matter of fact the position may shift along the axis, being dependent upon grid voltage and accelerating potential as well as cathode emission. It is this crossover point which is usually fo-

cused on the screen of cathode ray tubes when a small spot is required as in oscilloscope work or television.

It would seem that the more closely the crossover could be made to approach an infinitesimally small point the smaller the

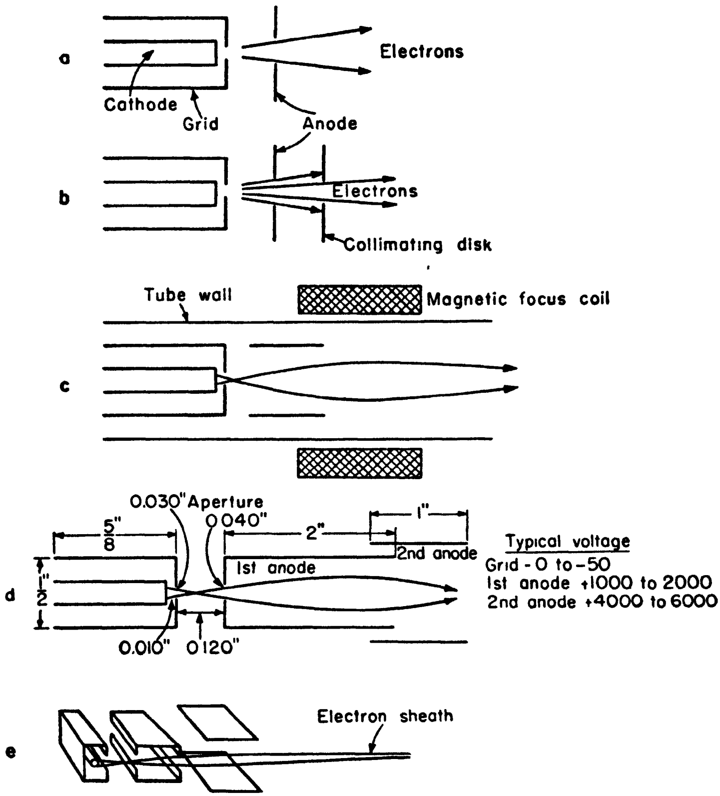


Fig. 13·16 Simple Types of Electron Guns. (3 in. high.)

focused spot would be. In actual practice the minimum diameter of this crossover point is determined by the fact that a practical construction giving such a perfect crossover is nearly impossible, and if it were possible other effects such as the mutual repulsion of the electrons would prohibit it for usable currents.

The maximum electron beam current density that can be attained has been found theoretically to be[4]

$$I = I_0\left(\frac{Ee}{300kT} + 1\right)\sin^2\phi \qquad (13\cdot15)$$

where I is the current density in the beam,
I_0 is the cathode current density,
E is the final acclerating potential in volts,
k is Boltzmann's constant 1.38047×10^{-16} erg per degree,
T is the absolute temperature,
ϕ is the half angle subtended by the beam.

In addition to the above limitations in beam concentration, parallel electron beams are subject to enlargement by divergence due to the mutual repulsion of the electrons. This enlargement may be determined from the relation.[5]

$$\Delta r = 3.04 \times 10^4 I V^{-3/2}\left(\frac{l}{D}\right)^2 r_0 \qquad (13\cdot16)$$

where Δr is the outward radial deflection of the outer electrons of a beam of diameter D (radius r_0), carrying I amperes accelerated by V volts along a distance l. The dimensions are all in centimeters.

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Chapter 14

THE UTILIZATION OF CHARGED PARTICLES

14·1 Required Properties of Receptors

The exact paths of charged particles are not directly visible in an electronic experiment or device using high vacuum. There is little value to an electron beam carrying a highly magnified electron image or to a beam deflected by a potential being measured if such intelligence cannot be converted to some useful indication. There are a number of methods by which one can obtain such conversion. For the most part they are based on the principle of applying the energy of the particles to some mechanism which will give a satisfactorily detailed visual indication of their presence.

In some of these methods the incidence of the particles gives rise immediately to visible light. In others a latent image may be formed which becomes visible upon subsequent treatment. When a knowledge of the intensity of electrons is desired, suitable electrodes and measuring equipment may be applied.

Which of the many methods used depends upon the conditions of the experiment. The choice may be dictated by ease of application. The material must satisfactorily withstand bombardment by particles at the voltage of the experiment. It must withstand evacuation, and its vapor pressure must not hinder the achievement of a good vacuum.

14·2 Fluorescent Materials

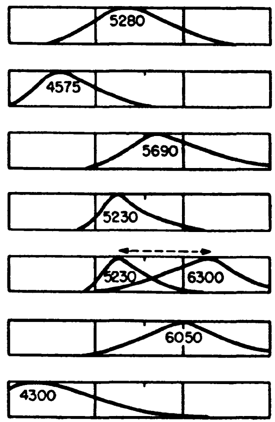
A large number of materials exhibit the property of luminescence.* Prominent among these are certain inorganic crystals

* Luminescence is usually considered to be composed of *fluorescence* having a very short decay period and *phosphorescence* having a much longer decay period.

TABLE I

CHARACTERISTICS OF PHOSPHORS COMMONLY USED IN ELECTRONICS

NO.	PHOSPHOR: BASE MATERIAL	PHOSPHOR: PER CENT ACTIVATOR	PHOSPHOR: TEMP °C	RELATIVE CATHODE-LUMINESCENCE: VISIBLE EFFICIENCY	RELATIVE CATHODE-LUMINESCENCE: PEAK ENERGY	SPECTRAL DISTRIBUTION CURVES (VIOLET, BLUE, GREEN, YELLOW, ORGE, RED; EYE MAX; 4000–7000A)	COLOR OF LUMINESCENCE	PHOSPHORESCENCE 0=VERY SHORT 5=VERY LONG	BEST Hg-LINE FOR ULTRAVIOLET EXCITATION: 3650A	BEST Hg-LINE FOR ULTRAVIOLET EXCITATION: 2537A
1	ZnS	0.01 Cu	900	230	340	5280	LIGHT YELLOW-GREEN	3	X	
2	ZnS	0.008 Ag	940	45	380	4575	LIGHT BLUE	2	X	
3	ZnS(50)CdS(50)	0.01 Ag	940	235	240	5690	LIGHT GREEN-YELLOW	0	X	
4	a-2ZnO•SiO$_2$	1 Mn	1250	100	100	5230	GREEN	1		X
5	$ZnO_w\ BeO_x\ SiO_{2\,y}$	Mn_z	1000-1300	50-110	15-110	5230 ←→ 6300	VARIABLE. GREEN TO RED-ORANGE	1-3		X
6	CdO•SiO$_2$	1 Mn	1150	45	—	6050	LIGHT YELLOW-ORANGE	4		X
7	CaO•WO$_3$	—	~1000	7	5	4300	PALE VIOLET	0		X



This table was compiled from a more extensive set of data published in *Proc. IRE,* Vol. 32, pp. 260–261, May 1944, by H. W. Leverenz, who has kindly given his permission for its use.

known as phosphors. From the large list of such materials, certain of them will be found to be mostly sensitive in the ultraviolet. Many are more efficient under X-radiation, and others may reach their emission maxima when bombarded by charged particles (cathodo-luminescence). The basic types most commonly used in electronics are listed in Table I, the silicates and sulphides being the most useful.

It is generally assumed that the ability of these crystalline phosphors to fluoresce is the result of strains produced in the crystals. These strains are induced by substitution in the crystal lattice of a small amount of "foreign" material called an activator. A fraction of a per cent of activator added in the making of a phosphor affects both the fluorescent efficiency, the persistence, and the color emitted by the material. For instance, zinc sulphide is usually activated by either copper or silver to the extent of a few hundredths of a per cent, and a wide range of colors and efficiencies may be achieved by proper use of these activators.

The light from a phosphor is usually emitted continuously over a wide range of the spectrum (Fig. 14·1) but is so peaked that it appears to be predominantly one color. Physical mixtures of phosphors may be used to achieve colors not available with any one alone.

The various phosphors have, in addition to their differences in stability and color of emission mentioned above, differences which must be considered when it is necessary to choose one for a particular experiment. Among these are differences in sensitivity to bombarding particles, both as to velocities and as to densities of the beams.

Measurements [1, 2, 3] on silicates, sulphides, and tungstates indicate that the light output from a fluorescent screen may be represented by a relation

$$L = Af(i)V^n \tag{14·1}$$

where L is the light output in arbitrary units, A is a constant, (i) is the current density, and V is the electron accelerating voltage.

With constant current density (i), the value of n is dependent upon the material and the manner of its application. For the

above phosphors n is approximately 2, being somewhat above that value for calcium tungstate and somewhat below it for the silicates.

In the above relation $f(i)$ is a rather complicated function, but a definite saturation or decrease in light output per unit current density with increasing current density is quite general with all the phosphors.

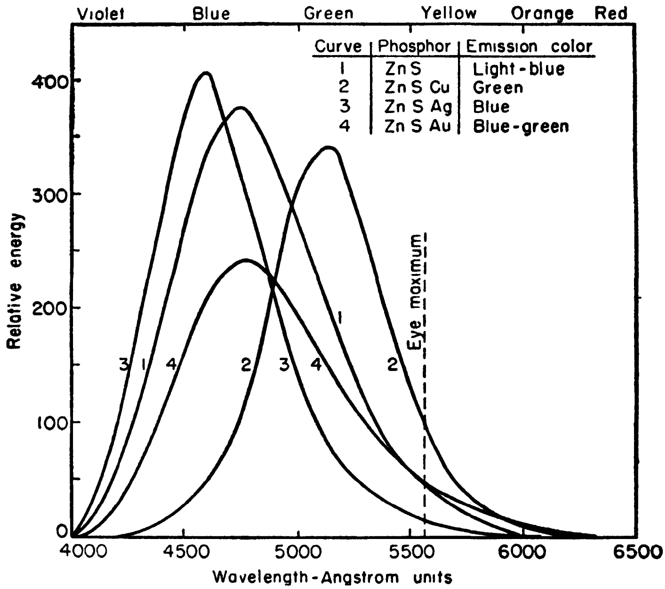


FIG. 14·1 Spectral Distribution Curves of Some Zinc-Sulphide Phosphors. (From H. W. Leverenz, *Proc. IRE,* Vol. 32, p. 259 [May 1944].)

In making studies of the light output with relation to the current density and accelerating potential it is found that data taken with a stationary beam of electrons differ somewhat from those obtained with a "scanning" beam. Also in relation 14·1 it is assumed that all the electrons in the beam arrive at the fluorescent screen with the velocity given them by potential V. The phosphor is an insulator and is commonly mounted on glass so that from the discussion on secondary emission in Section

12·6 it can be assumed that the problem of screen potentials is not simple.

Under special circumstances zinc sulphide and willemite have been made to fluoresce with electrons as slow as 3 or 4 equivalent volts.[4] However, the secondary emission properties of most screens are such that below 100 volts or so a secondary emission ratio of unity is not achieved, and the screen will charge up and refuse to accept electrons. Similarly, this same effect occurs for electron velocities of the order of 5 to 10 kv, and above, the value depending upon the phosphor.

If the potentials are higher the effect is referred to as "sticking" and the potential at which it occurs is the "sticking potential." Since the subject, together with some remedies, will be discussed at greater length in Chapter 16 the main features will merely be mentioned at this time.

The sticking potential of a screen is dependent, although only in a minor way, on the method by which the screen is applied and on the thickness of the layer. With very thin screens the supporting material is important because of the penetration of the electron beam. Numerous attempts have been made to include with the phosphors some material which would improve the secondary emission characteristics. With a few exceptions these attempts have not been very successful.

For applications in which particle velocities of a few kilovolts or more are used, great improvement may be obtained both in efficiency and in general quality of response by the application to the screen of a thin metal backing layer.[5, 6] A film of this kind may be made by evaporating aluminum on the screen. Ordinarily, evaporation of metal on such a rough surface gives a dull black deposit rather than a smooth metallic coating. A smooth coating may be achieved by first placing on the screen a thin film of organic material such as Formvar or nitrocellulose lacquer. The technique for this is described in Chapter 16. Such a metal film provides a conducting path for all incident electrons, thus eliminating sticking potential. Also the mirror-like form fitting coating turns back all light normally emitted in that direction and under some conditions can give rise to a 100 per cent increase in light output in the forward direction.

This scheme can also be used to good advantage with X-ray bombardment of fluorescent screens.

In addition to the described-above voltage and current characteristics each phosphor has its "build-up" and "decay" characteristics. Some have a rather long phosphorescent characteristic superimposed upon the fluorescence. This is indicated in Table I.

The distortion in the crystals which leads to improved fluorescence leads at the same time to a certain amount of instability since it may be relieved by excess temperatures or excessive bombardment of charged particles. The efficiency may also be reduced by physical maltreatment such as the grinding or ball-milling resorted to when it is necessary to reduce the size of the crystals. Table II shows the effect of ball-milling on the efficiency of willemite, one of the comparatively stable phosphors.

TABLE II

EFFECT OF BALL-MILLING ON PHOSPHOR EFFICIENCY

Hours Grinding	*% of Original Efficiency*
0	100
16	80
24	66
64	36

Data for α-Zn_2SiO_4 : Mn
From Leverenz, *J. Optical Soc. Am.*, Vol. 30, pp. 309–315, July 1940.

Artificial willemite is probably the most useful all-round phosphor for general electronic experimental work because of its stability, durability, and efficiency. The sulphides are also commonly used and offer a range of colors from which to choose. A number of manufacturers offer a wide variety of phosphors which are suitable for electronic work, and it is best to consult them if a material with a particular characteristic is required.

Fluorescent materials may be applied in a number of ways. Where uniformity of the screen is of no particular value to the experiment, fluorescent powder suspended in some fairly volatile material such as amyl acetate and ethyl acetate may be painted or daubed on the desired surface. This is useful often when only

some indication of the presence of the charged particles is needed. In such instances, thick layers of the material are not necessary. Most of the phosphors have rather persistent habits and, instead of its being difficult to apply them to the walls of the vacuum system, it is usually extremely difficult to rid the walls of the last traces once the material has been introduced.

The techniques for achieving uniform screens such as are necessary in cathode ray tubes and similar applications have been rather well developed. They consist of spraying, lacquer flowing, air settling, and liquid settling. An electrostatic precipitation method is becoming of more importance commercially. The technique is similar to that used in electrostatic dust precipitators. At present the method lacks the convenience and refinement achieved by the other techniques which are to be described.

Spraying is accomplished after first making a liquid suspension of the fluorescent powder in a suitable binder or carrier such as amyl alcohol. As in the case of oxide-coated cathodes, nitrocellulose lacquer is commonly used. It is thinned with amyl acetate or, for quicker drying, ethyl acetate. Any spray gun of a size suitable for the work at hand may be used. The spray pressure and nozzle adjustment will depend upon the type of work and the characteristics of the suspension, and individual adjustment is usually required for each job. It is desirable to arrange for agitation of the suspension in its container during the spraying. This may be mechanical or manual.

In the case of cathode ray tubes having a large screened area which must be reached through the small neck opening, special spray guns have been devised with extra long tilting nozzles. The tubes are preferably arranged in a holder in such a way that the bulb may be rotated as it is sprayed.

Considerable experimentation is necessary with any closed container of this kind because the air currents set up in the bulb by the high-pressure spray may cause the material to be deposited in unpredictable ways. Quite often the bulbs are heated to facilitate adherence of the wet powder as it hits the bulb face. The entire interior wall of such containers is usually coated to some extent during such a process, and it is necessary to remove this material for further processing. This is accomplished,

preferably while the material is still moist, or at least before it is baked, by means of long-handled scrapers and swabs.

When the spraying may be done on surfaces which are not confined to the interior of an envelope, straightforward methods are possible. With a little practice, considerable skill may be acquired in applying uniform surfaces. It is usually found helpful to use a brushing motion with the spray gun until the desired thickness is reached, and uniformity is also aided if the work can be rotated during the spraying process.

If a nitrocellulose lacquer is used, it must be oxidized before it is placed in the vacuum system or it will blacken under bombardment. This may be accomplished by baking it in air at a few hundred degrees centigrade. Temperatures a little over 200°C may be used, but the time required to remove the normal amount of lacquer will be much longer than if baking is carried out at higher temperatures. The higher limit is determined by the softening point of glass or other backing material used or the temperature at which the phosphor deteriorates. With sulphides, temperatures of 400° to 500°C should not be exceeded. Good circulation of air over the surface should be provided during the baking.

In lacquer flowing, the fluorescent material is suspended in nitrocellulose lacquer of thicker consistency than that used for spraying. A predetermined amount of this material is placed on the surface to be covered, care being taken that bubbles are eliminated. By tilting and rolling the surface the lacquer can be caused to flow out uniformly. The rotation and tilting should be continued to hold this uniformity until the material dries. In some cases uniformity may be maintained and the excess may be drained from the surface by inverting the work. Attempts to drain by placing the surface in a vertical plane nearly always lead to the screen's being thinner at the top and much thicker along the bottom edge.

A cathode ray tube screen may be applied in this manner rather quickly after some experience has been achieved. Just the right amount of material placed at exactly the right position on the window, coupled with a certain procedure of flowing and possibly accelerated drying by the application of heat, can produce a very neat-looking screen. The lacquer must be removed

by air baking just as in the case of the sprayed screen. Advantages of this method are that no special equipment is required and predetermined amounts of fluorescent material may be placed on a given area, resulting in a screen of predetermined thickness with practically no waste of fluorescent material.

In liquid settling the phosphor is allowed to settle out of a liquid suspension on to the surface which is to be coated. After the settling part of the operation is completed, it is necessary to remove the liquid. If the screen is being settled on the container itself, this may be done by tilting the container and decanting, or removal by vacuum siphoning may be resorted to. If the screen is being settled upon a flat, such as a glass disk or metal plate, the plate may be supported on a stand at some little distance from the bottom of the container, which may be fitted with a drain cock at some point below the plate being screened. In either case, great care must be taken during this step to insure that the settled screen, which will be in the moist state, does not slip during the tilting or become agitated by the action of the last amount of liquid draining from the surface.

Successful liquid settling is usually accomplished only after considerable experimenting. The particle size is an important factor. In practice success has been had [7] with suspensions having phosphor concentrations of 0.002 to 0.020 gram per milliliter of triple-distilled water. Ammonium carbonate is added to this, the amount being held constant with respect to the phosphor concentration at a ratio of about 12.5 grams of ammonium carbonate to each gram of phosphor of particle size 0.001 mm.

The liquid used for suspension is generally a weak electrolyte. In addition to the ammonium carbonate, carbamate, ammonium acetate, ammonium hydroxide, and weak acetic acid have been used. From previous trials the amount of suspension necessary for a suitable screen may be determined. There is no loss of material in this method, and in case a screen does not settle out properly, the container may be agitated to throw the phosphors into suspension for another attempt at settling.

The process is very sensitive to the temperature gradients set up near the walls of the container as a result of a difference between the surrounding temperature and that of the suspension. Circulating currents occur which may create peculiar patterns on

the finished screen. Building vibration or other agitation may also give rise to poor results. In addition to the "cook-book" nature of the technique required in the process, the time and space requirements place it at a disadvantage. However, rather close-packed smooth screens with comparatively good adherence are obtained by this method.

As the final moisture is evaporated from the screen material, release of that remaining within the screen structure tends to create minute explosions which cause pinholes. These are usually of a size that is not objectionable unless some magnification of the screen is desired. When very fine grain uniform screens are desired and complete coverage is of importance, the following procedure will give good results.

The phosphor is ball-milled in very pure amyl acetate until a particle size of 1 to 5 microns is obtained. This procedure will be accomplished only at the cost of some fluorescent efficiency, particularly with sulphides. The amyl acetate is removed by repeated ether washing, and the range of particles is separated by flotation. A suspension of the fresh ball-milled material is made in ether. The larger particles will drop out faster than the lighter ones, and by properly choosing the settling fraction, a good particle size may be obtained. This phosphor of proper size may be left in the ether and used as a stock suspension.

The concentration of the phosphor suspension may be controlled by the addition or subtraction of the ether. A few trial settlings will enable one to arrive at the concentration required for a given screen thickness. In some cases it will be possible to decant or drain away most of the ether, but for best results the last of the liquid should be allowed to evaporate slowly to complete dryness.

If excessive pinholes are encountered the addition of a non-reacting liquid having lower volatility than the ether may be added to the suspension. Benzene and dioxane are two such liquids. Usually much less than 1 per cent of such material is sufficient. The result in this case is that the ether evaporates first, leaving the phosphor wet with the slower evaporating material. This cuts down the tendency toward violent evaporation and thus reduces pinhole effects. Both the ether and the "retarder" must be kept extremely pure for high-quality screens.

The "air-settling" process of making fluorescent screens is sometimes known as the "dusting" method. Either term is equally descriptive since the method consists of creating a dust with the phosphor powders and allowing this dust to settle on the surface which it is desired to coat. As in liquid settling techniques the process is usually carried out in a confined chamber. Sometimes the tube which is to be screened is a suitable

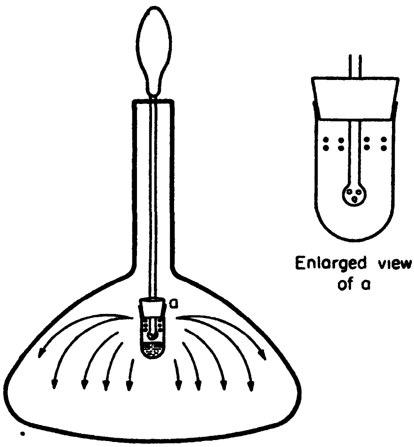


FIG. 14·2 Arrangement for Air-Settling Phosphors.

container; at other times, as in the screening of flat windows, an auxiliary chamber is used.

Certain of the phosphors are better than others for use in this method. Some of them, notably sulphides, may have a physical characteristic known as "free flowing." Such powders do not tend to clump or stick together and will flow like dry sand when the container is tilted or rolled. Free-flowing phosphors are best for the air-settling procedure.

A number of devices have been developed for creating the necessary dust conditions. Figure 14·2 shows one which is very satisfactory and quite easy to construct. A predetermined amount of phosphor is placed in the glass container, and the rubber stopper carrying the glass tube and rubber bulb is placed in position. When the rubber bulb is squeezed, air rushes from

the little holes at the end of the glass tube, creating violent agitation of the phosphor. The phosphor dust thus created issues from the small holes around the glass container and fills the chamber with phosphor which settles uniformly on the surface below. The small holes in the glass container should be of the order of 6 to 8 mils. They may be made by a technique whereby a hot pointed tungsten rod is repeatedly plunged into the glass.

To insure the uniformity of the screen deposited, it is desirable to have the chamber rotating independently of the settling device. There will be an optimum position of the dusting device in the chamber, which will depend upon the shape and size of the chamber and the characteristics of the dusting device. This technique is fairly economical in its use of phosphor since very little of the material sticks to the walls.

Although it is very easy to obtain excellent screens by this method with very little practice, the dusting part of the procedure is unfortunately a minor part. Such a screen exhibits no tendency to adhere to untreated glass, and some form of binder must be used. A number of binders are available, some of which are applied before and some after the air settling of the phosphors.

The old standby, nitrocellulose lacquer, here again is useful. In proper consistency, as found by experiment, it is either sprayed or flowed over the surface to be coated. When it has reached the right degree of tackiness, and this will depend upon the temperature of the surface and the characteristics of the lacquer, the air settling may be carried out. After the required amount of phosphor has been laid down, any excess material is cleaned away and the customary baking-out procedure is followed to remove the lacquer.

Another method makes use of a layer of sulphur to give adherence of the screen particles. The sulphur is applied by inverting the chamber so that the area to be coated is at the top. A small container, roughly the size of a thimble, is filled with carbon disulphide. This is ignited and by means of a suitable holder is placed up in the chamber several inches from the top (Fig. 14·3). As the carbon disulphide burns, the supply of oxygen in the chamber is diminished and sulphur is deposited on the upper surface. This layer is quite stable. An air-settled screen

deposited on the sulphur will be found to adhere quite satisfactorily. Excess powder may be tapped from the screen after a baking procedure similar to that used in the case of lacquer. If the sulphur were not removed by such baking it would poison the oxide-coated cathode.

In another method a solution of potassium sulphate (approximately 1 gram to 500 cc of water) is sloshed over the window

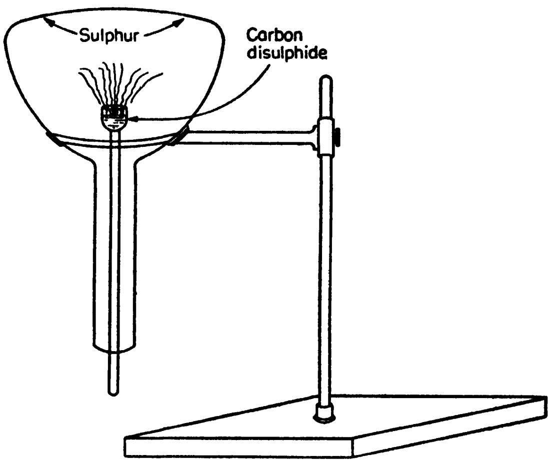


FIG. 14·3 Means of Depositing Sulphur.

to be coated. The excess is drained off and the surface dried and air-settled. Water vapor is then admitted to the chamber until moisture just begins to collect on the walls. Dry air is then passed through the chamber, after which the screen may be baked and further processed.

Both potassium and sodium silicates have been used as screen binders. In water solution they may be sprayed or flowed on the surface, and when they have reached the proper degree of drying, the phosphor is air-settled and the screen baked as before.

Water vapor alone at times will serve as a suitable binder. The surface preferably is exposed to water vapor before and after air settling and, although satisfactory binding is not accomplished every time, the process can be made to give suitable screens. In those techniques where water vapor is essential it

may easily be obtained by piping steam to the chamber through a hose or glass tube.

Another satisfactory water vapor source which allows considerable control makes use of the device shown in Fig. 14·4. A small glass thimble-shaped container similar to that used in preparing the sulphur binder is used. With a fine pointed torch the walls of the container are heated at several points to give rise to indentations so placed as to hold two parallel metal plates

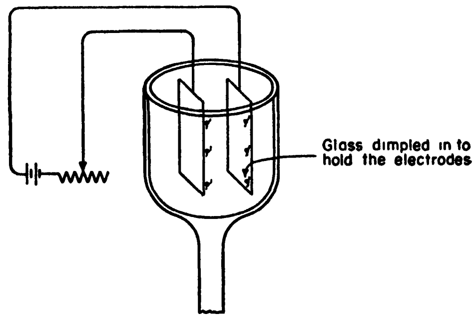


Fig. 14·4 Water Vapor Generator.

apart. Wires attached to these electrodes are connected to a source of potential which may be varied. When a small amount of water is placed in the thimble and a suitable potential applied, current will flow in the circuit and its passage through the water between the two plates will create sufficient heat to vaporize the water. This process is capable of being controlled very closely. Care should be taken that violent vaporization is avoided or particles of water will be thrown on to the screen, causing blemishes. Non-conducting water such as distilled water should not be used, of course.

A few general remarks may be made about the care and handling of fluorescent screens during processing. Whenever possible, it is best to keep them inverted since, except in the best of air-conditioned rooms, considerable dust is always floating around and contamination may result. It is always well to bake out a screen in air at as high a temperature as the stability of the phosphor and the glass will allow. This not only aids in remov-

ing materials which could cause trouble in vacuum but apparently also aids in stabilizing the adherence of the screen to its support. Sudden rushes of air across the screen should be avoided and vacuum should not be applied too suddenly. The porous nature of the screen means that there will be occluded air pockets which may erupt under sudden vacuum and blast away the phosphor.

14·3 Photographic Emulsions

That electronic bombardment can give rise to a latent image on a photographic emulsion has been known for many years.

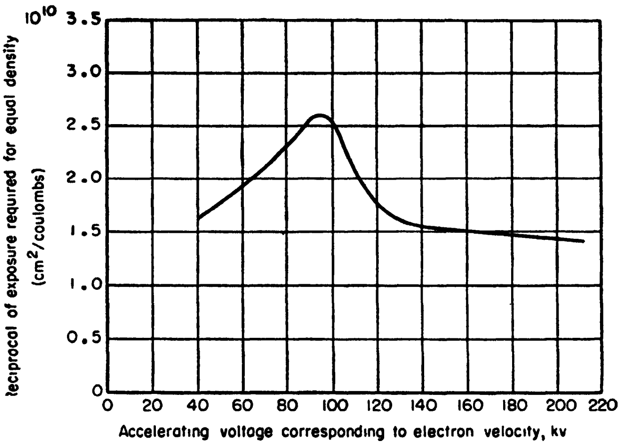


FIG. 14·5 Variation of Plate Sensitivity with Accelerating Voltage. (Eastman Medium Lantern Slide Plates.) (From Zworykin, Morton, Ramberg, Hillier, and Vance, *Electron Optics and the Electron Microscope*, p. 263, John Wiley & Sons, 1945.)

To accomplish this the electrons must have speeds of at least 25 equivalent volts, that is, they must have velocities given them by at least a 25-volt accelerating field. The sensitivity of an emulsion to electronic bombardment increases with the accelerating potential up to about 100 kv, where it goes through a maximum and then decreases (Fig. 14·5). The explanation advanced for the decrease is that at the higher voltages more and more of the electrons completely penetrate the emulsion. These elec-

trons contribute nothing to the exposure, and as their number increases the total exposure is diminished. The application of certain greases or oils to the emulsion increases the sensitivity, apparently due to the fluorescence of the oil by the electron bombardment.

Densitometry studies show that the reciprocity law holds in the case of electron bombardment, that is, the density of the image on the photographic plate depends upon the number of electrons striking the plate regardless of the time required for the exposure. It has been shown that the relative actinic effects of electrons at two specific velocities will be the same regardless of the image density at which such a comparison is measured.

For ordinary recording work where a great range of contrast is desired "contrast type" of emulsion has been found to be the most sensitive. For low values of blackening, however, X-ray film has been found to be slightly superior. Almost any photosensitive surface, however, whether glass plates, cut film, or sensitized paper, may be used for recording cathode rays. Depending upon the nature of the work, the individual worker may find different emulsions slightly superior. When linear measurements are to be made, glass-backed plates are in favor because of their physical stability. Celluloid and paper backing undergo considerable contraction and expansion during their exposure to vacuum and the subsequent wet processing of the emulsion. This difficulty can, of course, be obviated by the use of fiducial marks impressed on the emulsion at a suitable time during the processing.

The emulsion is usually made up of a layer of gelatin carrying the photosensitive material. Such a layer absorbs quantities of moisture, which slows down the pumping time of the system. Photosensitized papers are far worse in this respect than film or plates. Pre-evacuation or desiccation is commonly resorted to in order to eliminate much of the pumping time.

14·4 Color Centers

Certain alkali halide crystals become discolored when irradiated by X-rays or cathode rays. The effect can be shown most strikingly by using a crystal of one of the salts, say potassium chloride. Two electrodes, one pointed, are arranged as shown in

Fig. 14·6, the point being buried in the crystal, which should be several millimeters or a centimeter or so in extent. The electrodes should be arranged in a circuit which can supply 500 to 1000 volts. The crystal should be placed upon a base plate, which can be heated if desired to several hundred degrees centigrade. An asbestos or a metal plate heated by a torch or an electrical hot plate will suffice.

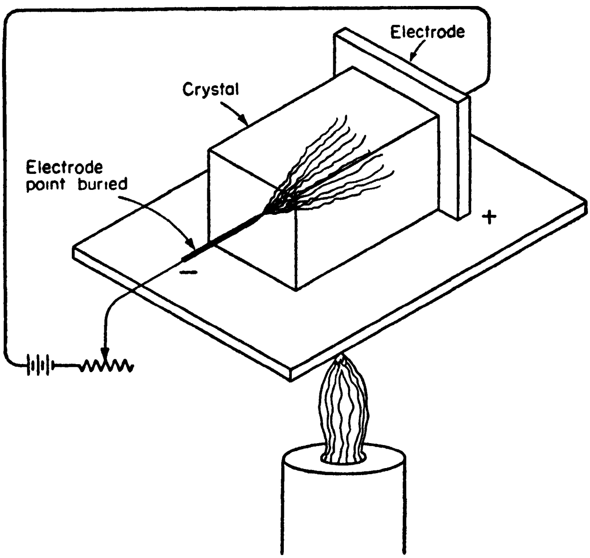


Fig. 14·6 Generation of Color Centers.

The point electrode should be made negative to the other by about 500 volts as the crystal is heated. Possibly a higher potential will be required, but at any rate with some combination of potential and temperature a dark line will appear to leave the point electrode and extend toward the anode, becoming more diffuse as it goes. Reversal of the electrodes causes the cloud of "color centers" to reverse and appear to go back into the point. The effect is very sensitive to temperature, and an increase of a few degrees is more effective than an increase of many volts.

Techniques have been developed for placing thin layers of these materials on the windows of cathode ray tubes, and under suitable conditions an electron beam moving over such a screen leaves a dark trace having a relatively long decay characteristic. Such tubes have become known as "dark-trace" tubes.

In such a tube the scanning electron beam leaves an actually darkened trace on the otherwise white screen, whereas in an ordinary cathode ray tube the fluorescent screen is actually a source of light. To make visual its information, the dark trace tube must be used with an auxiliary light source. This may be accomplished either in reflection or transmission. The image contrast obtainable with such a tube is limited by the degree of density obtainable in the trace. The coloring effect has build-up and decay characteristics quite dissimilar to those of fluorescent materials. The decay time, which is comparatively slow, may be accelerated by increasing the temperature or by bombarding the material with slow electrons. Some success has been had in varying the characteristics by the addition of small amounts of impurity in a manner similar to the activation of phosphors. Blackening may be increased up to a certain point by repeated bombardment of the same area, and in general the darker the trace the longer the time required for its removal.

The techniques involved in the preparation of such a screen are not too well known or understood. One method consists of evaporating the material onto the window from a small container suitably placed in the bulb and arranged to be heated under controlled conditions in a manner similar to that described in Section 9·7.

14·5 Temperature Effects

When a beam of charged particles falls upon a surface, it is necessary that the energy in the beam be dissipated. In the cases already described, the effects of fluorescence, the formation of a latent image, and crystal ionization, these phenomena predominated, but under some circumstances it is possible to arrange conditions so that the heating effect of the beam is utilized. Certain materials, some of which are known as temperature-sensitive paints, have the property of undergoing a pronounced color change at some critical temperature. In some cases this

process is self-restoring and repetitive; in other cases the change is permanent. When the conditions are right it is possible to create a local temperature at the point of impact of a beam sufficient to cause the color change.

One mixture which is quite sensitive is made by grinding together in a mortar approximately one part of cuprous iodide with two parts of mercuric iodide. This mixture may be suspended in water or some non-reacting oil or varnish such as nitrocellulose lacquer. Thin layers of this mixture may be painted on the surface (except aluminum) which is to be bombarded. At normal temperatures the color of the mixture is red, at about 70°C it becomes a chocolate brown, and it is black at 100°C. The colors change back upon cooling. Excess temperature causes a permanent yellow. The color change is extremely fast fundamentally but may be slowed down by the nature of the base upon which it is applied.

Another application making use of the heating effect is in connection with certain experimental tubes intended for television use. Into a mesh matrix is introduced a material which will incandesce when heated to the proper temperature. By auxiliary means the temperature of the matrix is maintained at a point just below the critical point for incandescence. At the point of impact of the beam the required extra heat is supplied to create an incandescent point light source.

14·6 Miscellaneous Receptors

In Section 14·3 it was pointed out that photographic emulsions are not sensitive to electrons having velocities under about 25 equivalent volts. In order to successfully record such slow electrons it is possible to make use of the fact that in some cases metal surfaces are affected by electron bombardment in such a way that they react differently when subsequently exposed to certain vapors or the areas are made evident by dusting with powder.[4, 8] By this means it has been possible to record electrons having velocities as low as 12 equivalent volts as well as those having high speeds. Metals having this sensitivity to electron bombardment are shown in Table III, together with notes on the vapors used in their development.

Among the other methods of utilizing charged particles is secondary emission, which has been discussed in earlier sections. Also electron beams have been used as catalysts to accelerate chemical reactions. Organic vapors may be decomposed when subjected to electrons. The subject of the production of X-rays is sufficiently large and well treated in the literature so that we need merely mention that an electron beam is used in all such applications.

TABLE III

METALS USED IN INDICATING ELECTRON BOMBARDMENT

Metal	Develop
Gold: plate, foil, leaf, or sputtered films	Several minutes in mercury vapor moisture condensed from breath
Silver: sheet or sputtered films	Ten to 20 minutes in iodine vapor
Zinc sheet	Hydrogen chloride vapor (H_2SO_4 on sodium chloride)
Brass, platinum, and nickel	Mercury vapor

Compiled from an article by P. H. Carr, *Rev. Sci. Instruments*, Vol. 1, pp. 711–743, December 1930.

In a variety of ways, beams of charged particles are used to complete circuits, for instance, in electronic switches and in indicating and triggering tubes of various kinds.

In many electronic experiments the current in a beam of charged particles is collected for measurement in what is known as a Faraday cage. This consists merely of an electrode in the form of a chamber of suitable size which is surrounded by baffles or shields; in some cases with suitable potentials, insuring that no charged particles are received other than those desired and that no charge is lost from the receiving chamber by such phenomena as secondary emission.

In addition to the various means of detecting charged particles discussed in this chapter, several techniques employed in the study of neutral molecular beams [9] might prove to be useful.

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Chapter 15

ASSEMBLY AND PROCESSING OF ELECTRONIC DEVICES

15·1 Mounting and Assembly Procedures

Most electronic devices, including radio and industrial tubes, consist of arrangements or assemblies of electrodes in vacuum, suitably spaced and insulated from each other and having suitable lead wires. Such an assembly is commonly called a "mount." The shape of the electrodes of such a mount is usually important, both from the standpoint of the electronic operation and also from the standpoint of assembly.

Small devices operating at comparatively low voltages are usually formed from such shapes as cylinders, plates, and helices, which are made of light gauge metal and wire. Nickel, Nichrome, 18-8 stainless steel, Monel, tantalum, molybdenum, and tungsten are commonly used. The thickness of such parts may run from less than a thousandth of an inch for ribbons to 30 or 40 thousandths of an inch for heavy plates and mounting rods.

From such metal a skillful mounter may fashion cylinders of various cross-sections, intricately shaped plates, disks, and cups which may be assembled into either standard or unorthodox assemblies for experimental purposes. When possible, it is very desirable to obtain standard parts from manufacturers of tubes or tube parts. This is particularly true of heaters, cathodes, seamless tubing, and aperture cups.

Devices requiring high voltage operation or high current dissipation will more frequently require heavier and thicker electrodes. They are customarily worked out in a machine shop where they may be given the proper contours and suitable rounding and polishing of the edges to meet the requirements of high voltage operation.

The tools required at the mounting bench depend somewhat upon the quantity of work to be turned out. A small vise, a variety of small pliers and cutters, and metal shears are essential. Although a pair of tin shears is desirable they are too large for most mounting work, and good shears similar to manicure scissors are necessary. A small bench type spot welder with numerous interchangeable electrodes is essential.

In electronic tube assemblies the accepted method of joining metal pieces is spot welding.[1] In this method high current is passed through the point of juncture of the two metals, the heat generated at the point fusing the metals thus making a weld. In high-production applications spot welders are carefully adjusted to the size and type of the work by mechanical or electronic timers. Although such equipment is to be desired, satisfactory results may be obtained in the experimental laboratory with a simple welder of 1 or 2 kw capacity.

Figure 15·1 shows those metals which may be welded together. When it is desirable to join two metals which do not weld readily it is sometimes possible to use an intermediate metal in the form of foil or ribbon to accomplish the purpose. For instance, copper braid may be attached to molybdenum if it is first bound at the point of contact with a nickel ribbon and the weld made through the nickel.

The necessity of hydrogen or vacuum firing a spot-welded structure to relieve the strains induced during the assembly was mentioned in Section 9·4. The spot welder can often be used to reduce strains by virtue of the localized heat it can produce. For instance, spot-welded joints can sometimes be relieved somewhat by going through the spot welding operation several times to heat the joint. Also when a support wire is suspected of being strained in tension it may be heated locally at a number of points along the wire by means of the welder electrodes. Such heating causes a small, but definite elongation of the wire.

Materials suitable for insulating purposes in vacuum include glass, mica, Mycalex, porcelain, and other commercial ceramics such as steatite. The ceramics must be formed at least roughly in the shape desired before firing, since they are usually amenable only to grinding afterwards. The tolerances which may be held through the firing process vary with the material and with the

shape of the structure. In certain instances a mil or less may be maintained. Commercial suppliers have a multitude of standard shapes and sizes and when possible it is most economical to adapt a structure to some existing ceramic part.

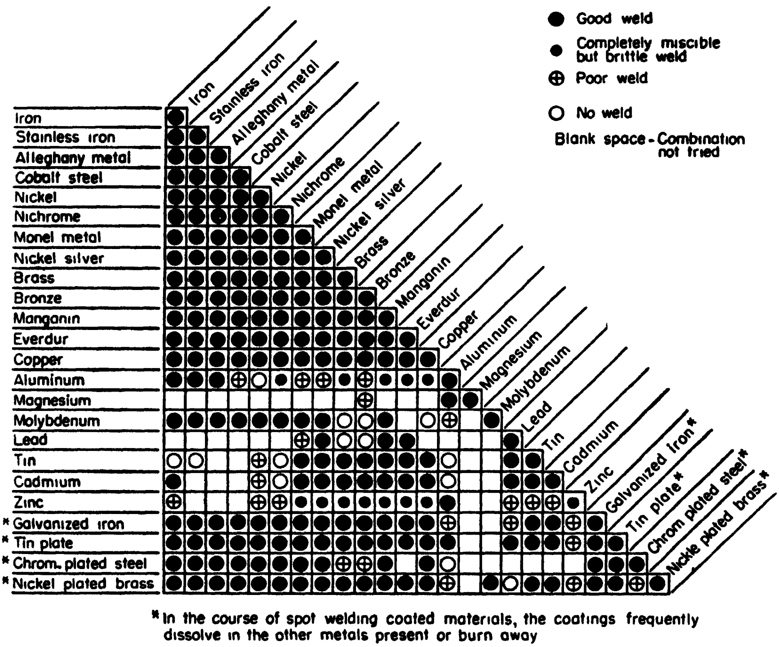


Fig. 15·1 Metal Combinations Which May Be Spot-welded. (From L. Ferguson, *The Welding Engineer*, July 1933 and August 1933.)

Talc (soapstone) (see Section 9·4) may be machined quite easily before firing and thus is very convenient for experimental work. Iron-free talc, "radio grade," should be used. Subsequent firing of the talc in air drives out its water and changes its structure, hardening it so that it can be worked only by grinding. This baking may be done at temperatures of 700°C up. The higher temperatures require less time but may give rise to warping. The time of firing varies, but a number of hours at about 1000°C may be sufficient for small pieces. The shrinkage that accompanies firing is not always uniform nor is it the same

in amount for all talc. It is usually desirable to fire a test piece which is the same shape and which is cut the same way from the same block of talc.

Mica is most conveniently utilized when it can be fitted with eyelets. They are available in various sizes, and devices for clamping the eyelet to mica are available in small size for hand operation as well as in various degrees of automatic complexity. The shank of the eyelet is split in such a way as to form four legs. In clamping to mica two of these legs are bent down to the mica whereas the other two are left upright and may be spot-welded to wires or other suitable structural parts.

In production methods mica is usually punched since cutting or drilling tends to tear the edges or open the laminations. When punching is not feasible it is usually desirable to drill mica while holding it clamped between metal templates or jigs. If the thickness is not too great, it may be shaped by cutting with a sharp pair of shears. If splitting is encountered it may be necessary to clamp the mica and use a sharp edge such as a razor blade to cut through the piece by drawing it repeatedly along the line to be cut.

After a piece is formed and fitted with eyelets it should be cleaned and hydrogen-fired before using. Firing at several hundred degrees centigrade is satisfactory. In many cases external leakage occurring along the surface may be minimized by means of surface treatment. One such treatment is merely the application of liquid milk of magnesia.

A very satisfactory means of assembling electrodes is by glass beads. Projections are placed on the electrodes in such relation that by using a hand torch with a small flame a piece of cane glass can be fused to a number of the projections, thus tying the electrodes together. A number of such beads are usually used to form a sufficiently rigid and strong structure.

It is not essential that the glass form a good glass-metal seal in this instance but only that it not shatter so much that it becomes too loose on the projection. Nonex or Pyrex may be used to bead molybdenum. The projections, usually offset wires, are quite often flattened on the ends which are to be embedded in the glass in order to anchor them more securely. Typical ap-

plications of various materials used in assembly are shown in Fig. 15·2.

The satisfactory operation of an electronic assembly depends upon a number of things, one of the most important being the accuracy of the construction and assembly of its parts. For instance, spacings between the cathode and grid are commonly set

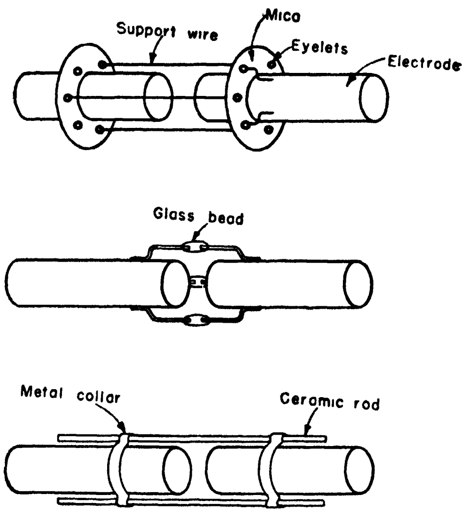


FIG. 15·2 Means of Supporting Electrode Assemblies.

to the nearest thousandth of an inch. Axial alignment of apertures and cylinders used in producing focused beams must be to similarly close tolerances. The sensitivity of deflection plates depends upon their spacing and the accuracy with which they are located.

In the assembly of electrodes, proper positioning is usually attained by means of fixtures and jigs. It is customary to build these very carefully since they determine the quality of the assembly to a large extent. These fixtures become quite a problem in some instances, since they must take on peculiar configurations to hold electrodes in place during the assembling, and then when the structure is finished they must be easily removable. In many instances they are built up of a number of parts

which are assembled by means of pins or dovetailing elements fitting together in such a way that they may be easily disassembled.

Many assemblies use cylinders which are provided with apertured disks either at the ends or in their interior. It is necessary that combinations of these cylinders and disks be held to close axial alignment. In the design of such an assembly it is customary to try to arrange the aperture diameters either to be equal or consecutively increasing toward the open or accessible

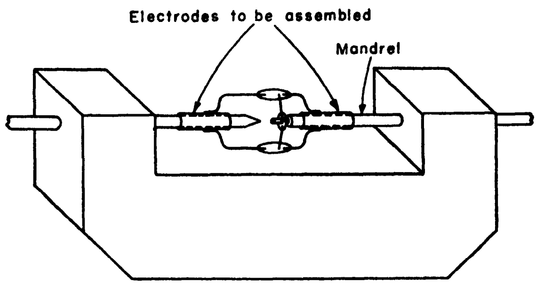


FIG. 15·3 Mounting Jig Useful in Assembling Electrodes.

end of the assembly. This makes it possible to hold them in place with a mandrel formed of rod, machined to have steps of different diameters, suitably placed to match the apertures. After the assembly has been tied together, as by spot welding or with glass beads, the mandrel may be withdrawn, leaving the resulting structure well aligned.

In some instances it is necessary to insert mandrels from each end of a proposed assembly, as when the aperture sizes decrease from both ends toward the center. In such a case a U-shaped holder such as shown in Fig. 15·3 is useful. This has provision for two mandrels which are carried in bearings in the two legs of the U, the bearings being very carefully machined to provide their being axially aligned. If the arrangement of consecutively diminishing apertures cannot be had, an oversize aperture may be held in place by means of a snug-fitting collar whose outside diameter fits the aperture and whose inside diameter fits the stepped mandrel. Figure 15·4 shows a typical assembly of an electrostatically deflected cathode ray tube.

Although fixtures of this nature hold the electrodes in good relation while they are being joined, the joining method may sometimes introduce considerable strain in the structure. Upon the removal of the fixture these strains may relieve themselves by some shifting of the parts, causing a loss in accuracy of assembly. If the strain relief does not occur at an earlier point the induction heating during the exhaust procedure may cause its occurrence.

As far as is possible strains due to spot-welded joints in the aligning structural members should be relieved during the as-

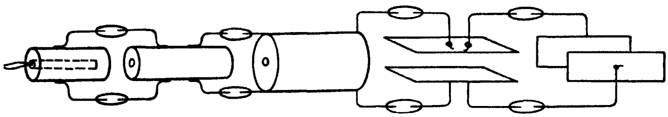


FIG. 15·4 Electrostatically Focused and Deflected Cathode Ray Tube Gun.

sembly operation. This may be done by high-frequency heating in hydrogen or vacuum prior to mounting on the stem. If necessary it may be accomplished while the parts are still mounted on the fixture. Another method of relieving strains of this nature is to repeat the weld a number of times with a much lower current adjustment of the spot welder. Although this subject of strains has been discussed earlier, the emphasis is justified since the matter is so often overlooked in practice.

There are times after a wire is welded in place when it would be desirable to have had it just a trifle longer. Some increase in length may be achieved by performing the low-current spot-welding operation upon it at one or more places along its length as mentioned somewhat earlier. In addition, a slight increase may be obtained by heavily denting the wire at a number of places with a pair of wire cutters. These schemes are often used to help straighten a mount on a stem when one mounting leg is too short.

We have already noted how contamination can affect the emission from cathodes and other sensitive surfaces. In addition it can cause surface leakage, discoloration of parts, and other such undesirable characteristics. The subject of parts cleaning was discussed in Section 9·4 in some detail. The high degree

of cleanliness required in working with electronic assemblies might at times appear exaggerated. However, in the manufacture of radio tubes the matter is quite serious, and great pains are taken to insure extreme cleanliness in the processing of the tubes. For instance, in some operations imperceptible perspiration from the hands of the assembly operators may play a major part in tube rejections. Consequently, cotton gloves must be worn in many stages of the assembly. Furthermore, it is essential that these gloves be lint-free. A thread or piece of lint may carbonize in such a way as to short two electrodes or contribute to noise or in some other way interfere with the normal operation of the tube.

Before assembly, metal parts should be degreased, preferably in some types of vapor degreaser. Such degreasers use solvents such as carbon tetrachloride, ethylene dichloride, or trichloroethylene. The material is usually arranged in a closed sheet metal tank containing a number of separate compartments. The solvent is heated and the vapor is caused to condense in the compartment used for the final cleaning. The parts are usually first immersed in the compartment farthest from this purified region and then successively in the cleaner solvent.

When such equipment is not available a common solvent such as carbon tetrachloride or ether is quite satisfactory. It should be kept in mind, however, that dissolved grease is distributed in such solvents uniformly and that immersion of a part in a solvent containing some grease is a sure means of providing a uniform but complete film of grease over the entire surface of the part. Thus good technique requires either that several baths in clean solvent be given or that the process consist of flushing the clean solvent over the part, thus washing away the impurities.

After degreasing with solvent, it is usually customary to remove by hydrogen firing any remaining materials which might cause trouble in the vacuum system. Ordinary dried hydrogen is suitable for most materials, but for exceptions such as stainless steel, Mu-metal, and such critical materials it is essential that the hydrogen used be extremely dry. It may be dried by passing it through a suitable line of driers which may consist of commercial drying compounds, liquid air traps, or hot copper

chips. It should be emphasized that tantalum should be hydrogen-fired with great care as it disintegrates under this treatment.

When an electrode mount is finally assembled and ready to be sealed into the envelope, it is common practice to wash it in boiling water or hot weak acetic acid and then dip it in alcohol to facilitate drying. Oxide-coated cathodes may be included in the washing if the oxide coating is the type suspended in lacquer. If the washed mount cannot be used right away it should be stored in a warm dry cabinet.

An assembly which is to be contained in an envelope and sealed off after evacuation is first mounted on a support containing the necessary lead wires. This support consists of either a flared cylinder or a disk which is provided with the necessary number of insulated leads and support wires. In the ordinary type of radio tube the support is called a stem and is made of a short cylinder of glass. One end of the cylinder is flared and the other end is provided with a press through which the lead wires are sealed. This stem is provided with a small exhaust tubulation which is fused in sealing off the tube.

In more recent types of tubes this arrangement has changed somewhat and consists of a disk with the lead wires circularly arranged around the exhaust tubulation which is centrally located. This arrangement is known as a header. Glass headers are available for use with glass envelopes and metal headers for metal-enclosed tubes. Figure 15·5 shows typical stems and headers.

Those wires which are carried through the press and are intended primarily for electrical purposes are called "leads." Those which are ended in the press and are used for mounting purposes are called "spuds" or "dummies." An assembly of electrodes may be completed and then mounted on a stem or it may be built up using the stem wires as part of the basic structure. It is essential that those leads carrying high current, such as filament leads, be of such a size that they will not be overheated and crack the stems. Care should also be taken that excessive potentials are not applied between leads or conduction may take place through the glass and result in breakdown.

Although a skilled glassblower can "seal in" mounts on headers, it is somewhat difficult because the many metal-glass seals must be heated quite high in order to get the header glass hot enough to make the seal to the tube. Automatic equipment with

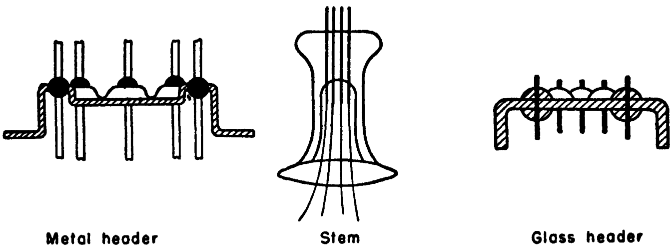


FIG. 15·5 Headers and Stems for Electron Tubes.

controlled preheating and annealing is the more practical way of handling headers.

In most experimental tubes headers have no particular advantage over flared stems and the latter are used. Figure 15·6 shows the method of sealing a mount on a stem into a piece of tubing. The exhaust tubulation may or may not be required,

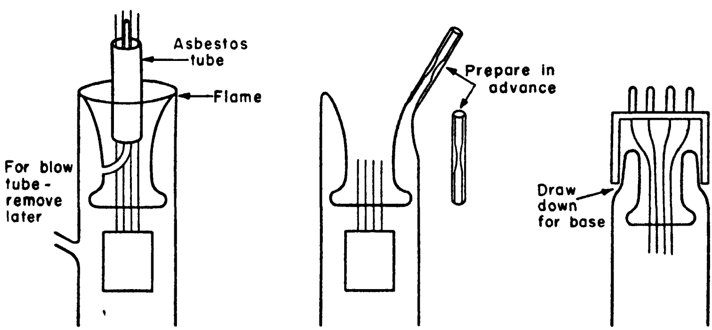


FIG. 15·6 Techniques for Sealing in Stems.

but, if so, it is thickened in advance and sealed to the tube while the rest of the seal is still hot. If a base is to be provided later the tube may be drawn slightly to give it a neck which will fit into the base. Line up of the mount in the tube

can be manually adjusted while the glass is still hot from making the seal.

For sealed-off tubes it is customary to provide a getter to clean up the final gas during processing. If this is to be flashed by high frequency, the getter pellet is usually mounted on a flag which is suspended from one of the leads in such a way that the flashed getter material will not cause leakage along important insulator surfaces. If the type of getter which is used requires passage of current through it in flashing, it may be mounted

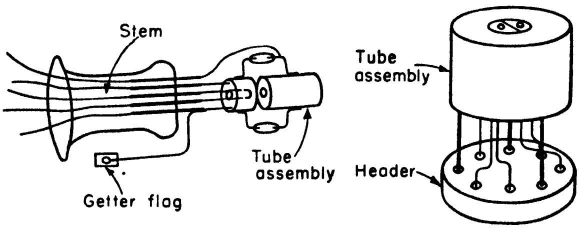


FIG. 15·7 Typical Tube Mounts.

between two leads which will tolerate being shorted electrically during the processing. After the getter is flashed, sufficient current may be passed to burn out this circuit, and thus relieve the short. Figure 15·7 shows typical assemblies ready to be sealed in.

15·2 Exhaust Procedure

When an electronic device consists of a tube which is to be evacuated and sealed off, there are a number of processing possibilities. If the tube is sufficiently large to require full pump capacity it is usually supported on an iron stand above the exhaust table. A bake-out oven is arranged to be lowered over the tube or otherwise placed in position when desired. The exhaust tube is sealed to the pump tube, which leads through the table top to the pumps below.

If the device is small compared to the available pumping capacity a number of them may be attached to what is known as a manifold. This is merely an enlarged tube leading from the pump and containing a number of ports along its side to which

are attached the tubes to be exhausted. A common oven is usually arranged which can be lowered over the entire manifold, and the tubes are processed simultaneously. This arrangement is used in the manufacture of special tubes, such as photocells, which do not lend themselves to mass production. Such tubes are usually light enough to be self-supporting, particularly when sealed to the manifold in an upright position.

Mass production procedures such as are encountered in the manufacture of small radio tubes now involve much automatic equipment. This equipment usually consists of a large rotary table with a number of ports or positions on its periphery. A mount on its stem together with a bulb or "envelope" are started in at one position. As the machine rotates, indexing from one position to the next, the tube moves around, being processed as it goes. Through the various stages it will be sealed into the envelope, baked, exhausted, and outgassed by induction. In addition, the electrical components will be tested for shorts, the cathode will be given its preliminary activation, and finally, after sealing-off, the getter will be flashed and the tube may be based. Machines are available which do all of these operations or any of the various steps.

Experimental procedure for the most part makes use of glass for envelopes and stems since this material is more flexible in its applications and requires less power equipment for ordinary shaping. In metal tubes, the electrodes are mounted on metal headers. In one process these headers are electrically welded to the envelopes and then processed, the exhaust being conducted through thin-walled metal tubulations.

In another process small glass bell jars are used. By means of exterior magnets, the metal envelope is held to the upper wall of the bell jar. No exhaust tube is needed as the entire bell jar is exhausted. At the proper place in the exhaust procedure the envelope is dropped over the mount and comes to rest in a groove in the header, which is filled with molten solder. The solder is then allowed to cool and a vacuum is thus maintained in the tube.

REFERENCE

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Chapter 16

MISCELLANEOUS HINTS AND TECHNIQUES

16·1 High-Voltage Notes

(*a*) *Materials.* In preceding chapters the properties of many materials, both conductors and insulators, which are used in vacuum work have been discussed. When these materials are to be used in connection with high voltages, such as one kilovolt or higher, their characteristics must be examined more closely to insure satisfactory operation. In Section 9·10, in connection with sputtering, we saw that at low gas pressures different materials responded quite differently to the application of high voltage. It is equally true that in a good vacuum various metals behave differently when exposed to extremely high voltage. This was pointed out in Section 12·3, where we discussed the emission of charged particles from points subjected to high fields.

In many applications it is necessary that metallic electrodes give stable operation under high field conditions, and here the choice of metal may be quite important. Table I of Chapter 12 shows the flashover potentials for a number of metals. It is seen that brass is low compared to metals such as chrome steel. Very good results are obtained, however, when the part is made of highly polished brass and then given a bright chromium plating. There are times when manufacture by this method may be more practical than using the harder materials. For instance, 18-8 stainless steel has fairly good non-magnetic properties and a high flashover point but is somewhat difficult to machine and polish.

In the fashioning of electrodes which are to be used under high voltage operation it is desirable that all surfaces exposed to high fields be well polished. In addition to this there should be no sharp edges or corners and abrupt changes of contour should be avoided. These requirements are particularly true of

surface irregularities of a convex nature. Those of a concave nature tend to be self-shielding. This should not be relied upon too extensively, however, for such concave irregularities as corners or cracks have a tendency to contain contaminating materials which are not easily cleaned out because of the inaccessibility.

Whereas, in low-voltage application, electrodes could be made of thin metals without regard to the radii of the exposed parts, assemblies for high-voltage applications are characterized by a more massive appearance. Edges are rounded and polished or if made of light metals the edges are spun over to achieve the desired radii for minimizing the field.

One of the difficulties attending the use of high-voltage equipment is that corona is formed along the conductors in and from the power supply. The wide use of X-ray equipment has led to the development of rubber-covered cables, usually enclosed in a braided metal sheath which may be grounded. In standard installations such rubber-covered cable is quite satisfactory, but in experimental set-ups, where connections may be temporary and not necessarily of the best design, trouble may be experienced due to exposed areas of the rubber building up isolated regions of charge which break over to ground from time to time.

At times such rubber-covered cable will be found quite satisfactory. In other instances it may be desirable or even necessary that bare conductors be used, the necessary insulation to ground being provided by sufficient air space around the conductor. Figure 16·1 shows the effect of conductor diameter upon corona loss at various potentials. The curves show the loss from one kilometer of the measured wire to a surrounding concentric collecting cylinder of one meter diameter. This information is for alternating current and is based on smooth surfaces. It may be partially or completely nullified by the presence of dust, abrasion, or other factors contributing to field emission.

Flexible metal tubing which is available in lengths sufficient for most laboratory applications makes very useful flexible high-voltage conductors. It is available in a wide range of diameters and may be obtained with or without a close-fitting woven protective sheath. It usually will be found that the fine wires used

in forming this sheath will eventually break and the resulting sharp points will give rise to corona.

It might be thought that without this sheath the individual convolutions of such tubing would be conducive to corona. Actually this is not the case, as the normally close spacings of

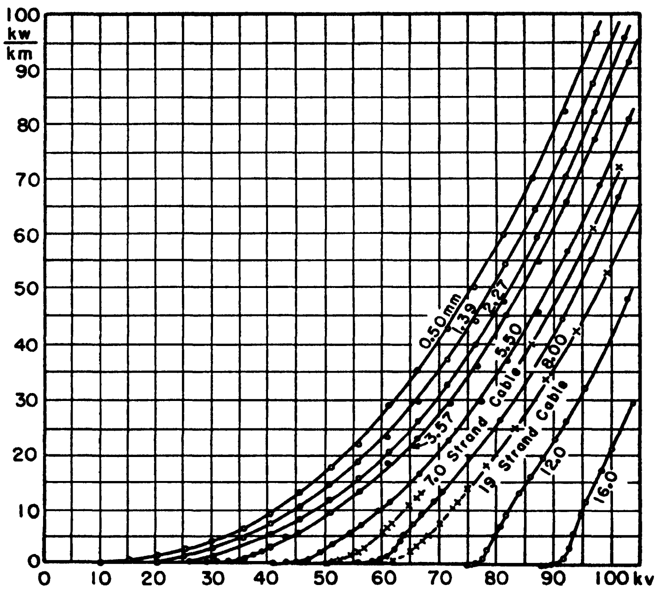


FIG. 16·1 Dependence of Corona Loss upon Voltage for Line Conductors of Various Wire Sizes (From Schwaiger and Sorensen, *Theory of Dielectrics*, p 290, Wiley & Sons, 1932)

the convolutions gives rise to a satisfactory smoothing out of the overall contour. However, if such tubing is bent sharply and the space between convolutions increased accordingly, the self-shielding effect is lost and corona will result at much lower voltages. Such tubing being hollow allows the threading through it of one or more conductors which are thus shielded from high fields.

Flexibility of a high-voltage lead may be obtained in still another fashion. A number of beads are formed either of metal or of some material coated with metal. These are strung on the

conductor carrying the high potential. This gives an effective large radius of the conductor but retains flexibility.

Ordinary copper tubing is often used when the diameter required makes solid rod or wire undesirable. It is obtainable in a wide range of diameters and may be bent and shaped by observing a few precautions. Bends which are not too sharp may be made without much difficulty. When this is not the case and trouble is experienced with the tubing collapsing at the bend, it has been found useful to fill the tubing with soft solder or Wood's metal * or in some instances, sand. This will hold the shape of the tube while allowing it to be bent, and the filler can be removed easily later. Since copper will be found to harden during bending or other working it may be advisable to anneal it frequently during the working operation. It should be done in a non-oxidizing atmosphere. A hydrogen flame will prove useful for this work.

At times necessity may dictate for a high-voltage lead or connector a particular shape which is bad from the standpoint of corona. In such instances it is often quite possible to add some superstructure such as a ring, a spherical cap, or an enclosing cylinder which may, by virtue of its configuration, minimize corona, and at the same time shield the irregularly shaped equipment contained within it.

Although it was not necessary to give much thought to insulator design for low-voltage application, it is extremely important in high-voltage work. Corona effects accompanying high-voltage applications in air or gas are not encountered in vacuum work. On the other hand, care must be exercised to insure that high fields do not ruin the vacuum by causing uncontrolled field emission with resulting violent gas evolution.

Insulators must be designed to eliminate volume and surface creepage, to furnish the necessary physical strength to the structure, and to provide sufficient dielectric strength to withstand the applied voltage. The customary rules of high-voltage insulator design may be followed to a great extent except that more attention must be given to surface effects. Although such insulators

* Woods metal consists of 50 per cent Bi, 12.5 per cent Cd, 12.5 per cent Sn, and 25 per cent Pb. It melts at about 60°C.

as glass and Mycalex may be operated at higher potentials if left with their natural glazed finish, the results are likely to be inconsistent. Consistency may be achieved by sandblasting or uniformly etching all exposed surfaces, although this will result in surface breakdown potentials a little under the best which might be obtained with some surfaces.

There are a few basic considerations to be remembered in the use of insulators either in air or in vacuum. In Fig. 16·2*a* the field between the two plate electrodes is given by V/d. Assume

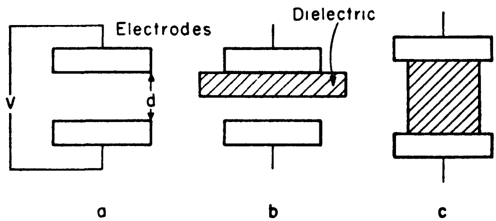


FIG. 16·2 Substitution of Solid Dielectrics in an Air Condenser.

V to be 30,000 volts and d to be 1 cm. The field is then 30,000 volts per cm. Although this is close to the limit, assume that in air there will not be a breakdown between the plates. Neither will there be one in vacuum. Now an insulating sheet having a thickness of ½ cm and dielectric constant of 5 is inserted in the space between the electrodes as in Fig. 16·2*b*. In effect this gives two condensers in series, and the voltage will be divided in a manner inversely proportional to the capacities. The insulator will get ⅙ or 5000 volts and the air space will get ⅚ or 25,000 volts. The air gap being now only ½ cm, the field will be 50,000 volts per cm. Such a field will certainly cause flashover in air and possibly in vacuum if the materials are not good.

If the insulator must give mechanical support and thus is made to fill the interelectrode space completely as in Fig. 16·2*c*, the limiting factor will probably be surface leakage rather than rupture of the dielectric, and the first thought will be to increase the leakage path. It may be increased by some variation of one of the methods shown in Fig. 16·3. The flanges in the design of

Fig. 16·3*a* approximate the arrangement of Fig. 16·2*b* and so contribute that weakness to the design. In the design of Fig. 16·3*b*, high fields occur at the edges of the plates, and rounding of the corner merely changes the shape toward that of Fig. 16·3*a* by providing an air gap. Figure 16·3*c* shows an insulating support giving long leakage path but allowing the metal electrodes to determine the breakdown potential.

When a supporting insulator must be used between electrodes whether in vacuum or air, the dielectric strength of the insulator

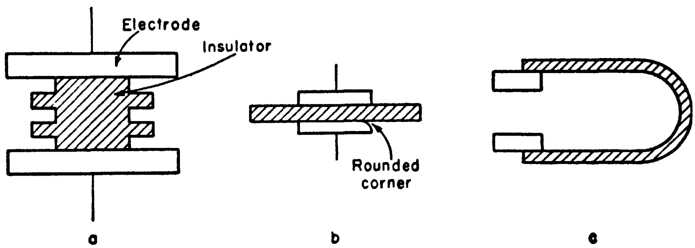


FIG. 16·3 Supporting Insulators of Various Shapes.

must be sufficient to withstand the applied fields, the surface paths must be sufficiently great to keep leakage under control, and as much as possible the insulator should be shaped so that its surface falls in parallel with the lines of force between the electrodes. Figure 16·4 shows how these principles are applied to the design of an insulator to support the center electrode of a unipotential electrostatic lens. The lines of force between the center electrode and the two outer ones are shown at the left, and the cross-section of the insulating washer at the right shows how its shape conforms. Other means of providing high-voltage insulation for this arrangement of electrodes is shown in Fig. 16·5.

Spark plugs form a cheap and convenient means of insulating for high voltage when the vacuum wall is metal. Also when the exposed rubber can be tolerated some of the forms of antivibration mountings in which the rubber is well bonded to the metal supports may be used.[1]

The scheme of Fig. 16·3*c* is shown applied to an insulator for

an electron gun in Fig. 16·6*a* whereas Fig. 16·6*b* is an application of Fig. 16·3*a*.

In some d-c insulator applications, charges will build up on the insulator surface until they reach a limiting value at which

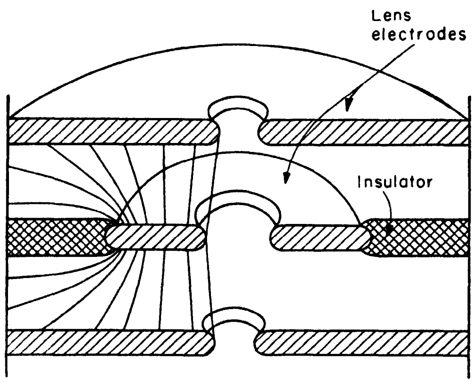


Fig. 16·4 Electrostatic Lines of Force in Lens and Conforming Insulator Design.

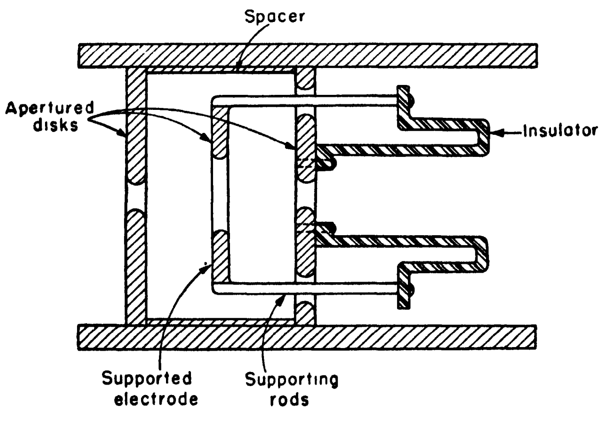


Fig. 16·5 Insulating Lens Support.

they discharge with a snap that may set up radio frequency oscillations in associated circuits. Such charges as well as non-uniform leakage over insulator surfaces often distort electron optical images. For instance, the glass beads used in the construction of an electron gun may charge up and deflect the beam.

When the current capacity of the power supply will permit, such non-uniformities may be smoothed out by actually making the insulator slightly but uniformly conducting. High-resistance

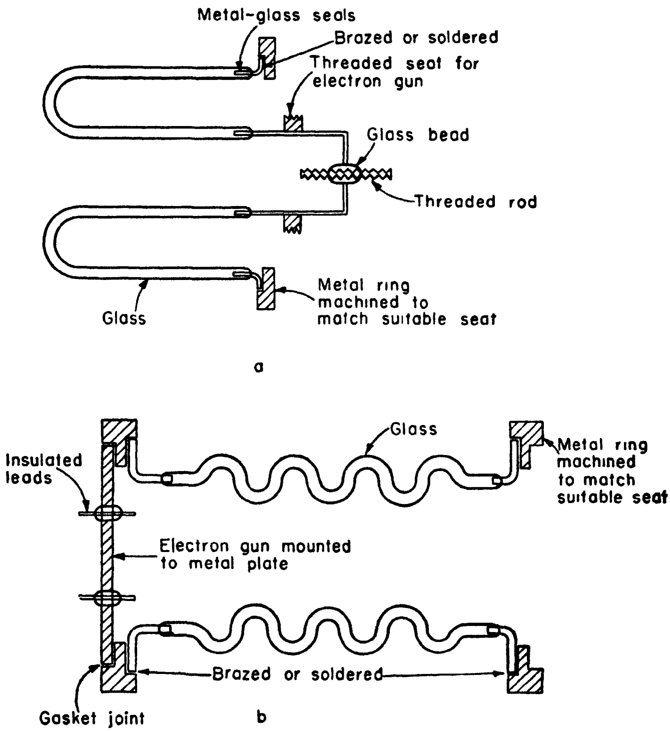


FIG. 16·6 Gun Insulator Designs.

paints such as Reskon * may be applied with good results to insulators under conditions permitting controlled leakage.

(*b*) *Sticking Potential.* When an insulating surface such as glass or a fluorescent screen is bombarded by electrons, it will receive these electrons only if conditions are such that for every electron received an electron is lost by some means. If the excess electrons are not removed in some fashion the space charge which is built up prevents further electrons from reaching the

* General Electric Co., Pittsfield, Mass.

surface. The potential corresponding to the electron velocity at which this saturation effect occurs is known as the "sticking potential." In the case of fluorescent screens discussed in Chapter 14 its presence is made evident when no increase in brightness is forthcoming as a result of increased beam potentials. (A similar effect is obtained in very low voltage work where, under certain conditions, it is impossible to make the electrons arrive at the insulator.)

The phenomenon underlying this situation is secondary emission. In Section 12·6 we saw that when a material is bombarded by electrons there is a certain range of accelerating voltages over which there may be a secondary emission ratio of greater than one. Above or below this range the material will not give rise to a secondary electron for every impinging primary electron, and as a result a space charge is developed.

A number of ways are available for eliminating or at least raising the effective value of this sticking potential. In the case of phosphors there are some substances, for example, certain beryllium compounds, which may be introduced into the screen to give it increased secondary emitting qualities. Sometimes the screen may be arranged to have greater conduction. The phosphor may be coated thinly upon a support which either is capable of conducting away the charge or may give rise to sufficient secondaries as a result of those electrons arriving at it after penetration of the screen.

Assuming the main electron beam to be of such high velocity that it does not give rise to sufficient secondaries, the space charge may be removed by bombarding the insulating surface with an auxiliary beam of lower potential. This auxiliary beam must be adjusted in current and voltage to the conditions where it furnishes the secondaries both for itself and the high-voltage beam. A trace of gas may be very effective in preventing the formation of the space charge, but since the presence of gas may not be in the interest of high-voltage operation this method is not of too great value practically.

In Section 14·2, on fluorescent materials, the use of a thin metallic film was mentioned. This technique is useful when accelerating potentials above 5 kv are to be used. A thin film of metal such as aluminum is deposited on the fluorescent screen.

The electron beam must penetrate this film to reach the fluorescent screen, and ordinarily an effective retardation of 2 to 3 kv results.

A cross-section of the fluorescent screen provided with such a thin film is shown in Fig. 16·7. The film is best applied to the screen by evaporation. Ordinarily, when aluminum is evaporated upon a powdery surface, such as the fluorescent screen, it does not form a continuous metal layer but is laid down in the form of a gray-black powder which does not have the conducting properties of metal. However, if a thin organic membrane

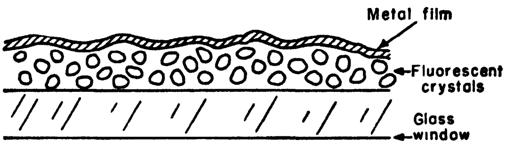


FIG. 16·7 Cross-Section of Metal-Filmed Fluorescent Screen.

such as collodion or Formvar is first applied to the screen it will present a sufficiently smooth surface to the evaporated aluminum that a metallic coating will result. The membrane presents no serious obstacle to the penetration of the electron beam and may usually be disregarded in processing. However, if an excess is used it should be removed after the metal has been deposited by baking in air to avoid discoloration and possible decrease in the quality of fluorescent screen.

The next section will describe the technique of forming thin films on water. If such a thin film, say of Formvar, is carefully transferred on a wire ring to the surface of the screen and then exposed to water or water vapor, it will cling to the crystals and will pull tight across the undercuts and pits. When dry, the wire ring is easily removed and the remaining film provides a smooth surface for the evaporated metal.

By another method * which is useful when small-necked tubes are to be coated moisture is allowed just to fill in the regions between the particles. The whole tube is then cooled until the water freezes. A thin solution of cooled Formvar is flowed over the iced screen, leaving a thin film which will cling to the par-

* V. Schaefer, U. S. Patents 2374310 and 2374311.

ticles and provide the surface for evaporation. The moisture can be removed through the Formvar film easily. The Formvar film presents no penetration problem to the electron beam and in fact is usually decomposed during subsequent processing.

The effect of such a thin metallic layer is shown in Fig. 16·8, sketched from experimental data.

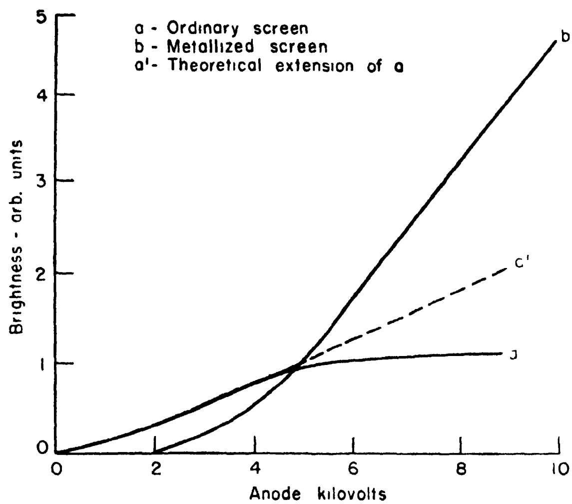


FIG. 16·8 Brightness Curves for Ordinary and Metal-Filmed Screens.

When the particles of phosphor are caused to fluoresce, the emitted light is given off from each crystal in all directions. In a normal screen this results in half of the light's being emitted in a backward direction. The form-fitting aluminum film functions for each crystal as a tiny reflector, and light emitted in the backward direction is thrown forward with a resulting increase in light output. There are other advantages in using such a thin metal film. It improves the contrast obtainable on the screen by eliminating reflections from the bulb walls. It also provides means whereby all impinging electrons are either conducted away or compensated for by the emission of sufficient secondaries, thus removing the sticking potential limitations on brightness.

(c) *After-acceleration.* "After-acceleration" (or "post-acceleration") is the term applied to the procedure, whereby the final acceleration of an electron beam is accomplished after it has passed the deflection plates or coils of a cathode ray tube. Such acceleration is provided by applying potentials to one or more rings as shown in Fig. 16·9. In the simplest form such a ring

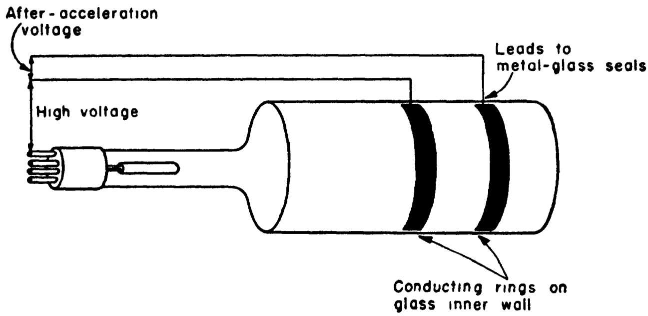


FIG. 16·9 Arrangement for After-Acceleration.

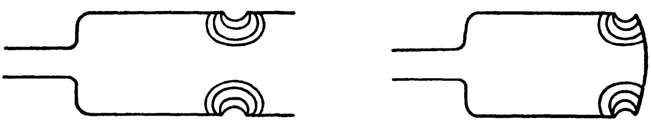


FIG. 16·10 Distortion Reduction in After-Acceleration by Use of Metal-Backed Phosphor.

may be made by leaving an uncoated ring on the otherwise conductively covered interior walls of the bulb. A lead must be provided in the bulb wall. Such a ring, together with the adjoining bulb coating, forms an electron lens. The distortion introduced by this lens effect becomes pronounced if after-acceleration of more than twice the anode potential is used.

The use of metal-covered screens gives a more favorable electrode geometry (Fig. 16·10), and with this arrangement it is possible to use after-acceleration potentials of five times the anode potential.

One of the reasons for using after-acceleration is in order that deflection of the beam may take place in a region of lower beam

velocity. Pierce [2] defines deflection sensibility of a cathode ray tube as the reciprocal of the change in deflection voltage or current required to move the spot one spot diameter on the screen. He points out that after-acceleration makes possible improvement in deflection sensibility in electrostatic deflection, but not in magnetic deflection.

Various schemes may be used to diminish leakage and sparking from the accelerating ring to the anode. The glass bulb may be softened and collapsed to form a ridge between the coatings or a number of auxiliary rings may be used to "grade" the potential between the electrodes.

16·2 Preparation of Thin Organic Films

Thin self-supporting organic films may often be made to serve in a useful capacity in electronic experiments. As discussed in the previous section, their use may make possible the application of metallic coatings to irregular surfaces such as fluorescent screens. They may also serve as supports for thin films of metal as in experiments involving the penetration of electrons through thin foils. Such organic films also serve as supports for specimens used in electronic microscopy and electron diffraction work.

Of the many films which have been studied, two have become most prominent, possibly by virtue of their utilization in the field of electron microscopy. They are the films made from collodion and from Formvar.*

In the use of either material, the film is caused to be floated on the surface of water. Subsequent operations are the same for each material, but the method of forming the thin film on water differs markedly. In either case, the operation is performed on a carefully cleaned water surface. Usually a shallow tray is carefully leveled and completely filled with water. Two straight rods, preferably of rectangular cross-section, may be used for cleaning. One of these rods is placed across the tray of water near one edge. If the tray is filled sufficiently, the water will wet this cleaning rod from side to side of the tray. If the rod is carefully moved in a direction normal to itself across to the

* Formvar (polyvinyl formal) 1595 is obtainable from the Shawinigan Products Corp., Empire State Building, New York, N. Y.

tray, surface dust and contamination will be swept before it. The second rod is then used to complete the same operation, and the water surface behind it will be found to be quite clean.

To form a collodion film, a drop of collodion of suitable concentration is placed on this clean surface. It will immediately spread outward in all directions, suddenly appearing to "freeze" as the solvent evaporates. The extreme edges of this film, now floating on the surface, will be found to be much thinner than the central region, and with experience an area of the desired thickness and fairly uniform quality may be chosen. If the collodion is too tacky, it will refuse to spread on the water and if it is too thin it will give films which are fragile and not self-supporting. Amyl acetate may be used as a thinner, and after some experience the consistency of the collodion may be controlled in a way that will give useful films.

It is not feasible to use the collodion technique for Formvar. To obtain a thin film of this material one first obtains a highly polished surface at least as large as the film to be formed. Good-quality glass plates are quite satisfactory for this purpose. Microscope slides are very convenient for small films. The plate should be carefully cleaned, for which a scouring or polishing agent such as Shamva * is excellent. Milk of magnesia may also be used successfully. Ordinary soap and water cleaning is usually not sufficient. The cleansing should be carried on under running water.

As the plate is cleaned and flushed under running water it should not be handled directly with the fingers, since a film of grease will immediately flow out from the point of contact with the fingers and thus ruin the effects of careful cleaning. The plate is next immersed in a solution of Formvar in ethylene dichloride, withdrawn, and immediately placed in a position so that the thickening due to drainage will be along an edge which is unimportant. The thickness of the film will depend upon the concentration of Formvar in the solution. Up to about 1000A (1 per cent solution †) the film thickness is linearly proportional

* Shamva is a metallographic polishing powder obtainable from the Golwynne Magnesite and Magnesium Corp., 420 Lexington Ave., New York, N. Y.

† One gram of Formvar to 100 cc of ethylene dichloride.

to the concentration. For greater concentrations the linearity fails as a result of viscosity effects, and thicker films result.

When the Formvar has dried upon the plate, a sharp-edged tool such as a knife blade should be scraped along the edges of the plate, thus breaking the continuity of the Formvar film over the edges. Next one edge of the plate is immersed in water as shown in Fig. 16·11. As water works between the film and the plate, the film should float off onto the surface of the water. The technique is not at all difficult to learn. If the film comes part way from the plate, but tends to stick occasionally, it may some-

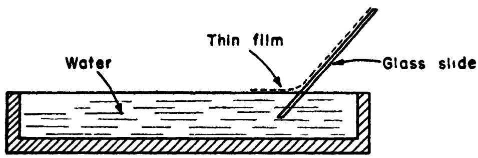


FIG. 16·11 Floating Formvar Film onto Water Surface.

times be possible to keep it moving by pulling gently on the film. Stubborn films also may be started sometimes by breathing upon them. The above technique may be used to obtain very large film areas having uniform thickness.

Once the film is floating on the water, subsequent operations depend somewhat on the use to which it is to be put. It may be removed in large areas by lowering over it a frame such as a ring provided with a suitable handle. The face of such a frame may be coated with rubber cement which may facilitate handling. Care must be taken that contact is made with the film completely around the frame. To lift the film from the water, the frame is slightly tilted into the water and then skidded up and away from the surface. In place of the rubber cement the portion of the film protruding beyond the film may be carefully lifted up and folded over the frame edge. This usually makes a sufficiently good bond.

Difficulty may be encountered if the film is not large enough to fill the frame completely since this space may allow water to run in on the top side. Also, in drying, the shrinkage along the unsupported edge of the film may cause its rupture. If the film

is very much larger than the ring the excess material may cause difficulty by folding back upon the main film and giving surfaces of multiple thicknesses.

This excess film outside the ring may be removed by a number of methods. With care, it may be clipped away with scissors. Usually the wet film tends to stick to the scissors' blades, however, and may pull a good film away from the frame. A small loop of fine wire arranged in a handle and heated by passing current through it is very useful in cutting through the film. It burns its way through the material, leaving a clean opening. Another method giving the same results uses the Formvar solvent vapor. This may be carried to the film by means of a rod or small brush dipped in the solvent or it may be fed through a capillary tubing attached to a small reservoir. Since the thin films are extremely sensitive to the solvent, great care must be used that the vapor does not cause deterioration of the main film.

Once the film is mounted on a ring and dried it may be used in a number of ways. The ring may be mounted in an evaporation apparatus in such a way that the film forms a support for evaporated material. Portions of the film may be transferred to smaller rings or to other surfaces by techniques corresponding to that used in lifting the film from water.

It is to some extent possible to cause a Formvar film to conform to irregular shapes. The material will soften when subjected to heat or moisture. For instance, if a film is picked up on the open mouth of a glass funnel and then exposed to moist warm air it is possible by means of slight air pressure applied through the funnel tube to blow the Formvar out into a hemispherical bubble. A method of achieving the form-fitting characteristic discussed in the application to fluorescent screens consists of laying the film in its ring down upon the fluorescent screen. In some instances "wetting" the film to the screen may be achieved by breathing upon it. If this fails a small amount of water may be carefully placed on top of the film. This water will cause the film to sag against the screen particles, and, by rolling and tilting the surface, thus shifting the water around, good adherence may be achieved over the entire area.

Another method of forming the Formvar film on rough surfaces, such as fluorescent screens, was described in the last section. It consists of wetting the screen with water, which is then frozen. Over this smooth layer of ice precooled Formvar is poured. The excess is poured off, and the result is a film of Formvar over the ice. The ice may now be allowed to melt. The moisture permeates the Formvar and evaporates, leaving the film in contact with the screen.

16·3 Metal-Glass Combinations Commercially Available

In addition to metal-glass seals in glass systems it is sometimes desirable to provide a metal system with a glass window. Commercially available are a number of press-molded glass shapes, provided with flanges of thin metal partially buried in the glass, which may be soldered or welded to the main structure.

Commercial techniques have also been worked out by which glass may be copper-coated in such a manner that the copper may be soldered to and the entire assembly made vacuum-tight. A number of standard glass shapes are available with this treatment ready for soldering.

16·4 Preparation of Small Apertures

For the most part the apertures used in electronic devices are sufficiently large so that ordinary drilling or machining operations may be used. In some instances, however, extremely small apertures are desired for masking purposes or for some other reason. Drills are available in sizes down to a few thousandths of an inch. For smaller apertures some other method must be used. The successful forming of such a small aperture depends to a large extent upon the material. Molybdenum is quite difficult to work, for instance, whereas platinum or tantalum are quite satisfactory, as is also copper.

As has been pointed out previously, for electron beam work the thickness of the material at the aperture should not be greater than the diameter. For a one mil aperture in a large diaphragm this would mean a rather fragile mount. For such an aperture a thicker material, say about 5 mils, may be used by the following method.

A rounded impression or dent is made at the point where the aperture is to be located. The material protruding on the back side of this dent is carefully ground away, giving an area of extreme thinness at the desired point while still leaving sufficient material around it. With a much sharper tool another dent is made and the protruding material is again removed. This time great care is taken to stop the grinding or honing just as the hole is seen to open. A fine reamer opens the hole to the required diameter.

After the reaming, a fine abrasive removes the ragged shoulder built up around the aperture. One advantage of platinum for small aperture work is that cleaning may be accomplished by heat or acid treatments which could not be withstood by other metals.

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1. H. Herne, *J. Sci. Instruments,* Vol. 23, p. 244, October 1946.
2. J. R. Pierce, *Proc. IRE,* Vol. 29, p. 28, January 1941.

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